

ARPA ORDER NO. 3363

REPORT NO. 82-1

**DEVELOPMENT OF A TABLETOP MODEL
FOR THE GENERATION
OF AMORPHOUS/MICROCRYSTALLINE
METAL POWDERS**

FINAL TECHNICAL REPORT
TO
OFFICE OF NAVAL RESEARCH
CONTRACT NO. N00014-77-C-0373
JULY 1982

Principal Investigator:
John F. Mahoney
(213) 357-3201

DTIC
ELECT
OCT 21 1982
A



Phrasor Scientific, Inc.

1536 HIGHLAND AVENUE
DUARTE, CALIFORNIA 91010
TELEPHONE: (213) 357-3201

The views and conclusions contained in this report are those of Phrasor Scientific, Inc., and should not be interpreted as necessarily representing the official policies, either express or implied, of the Defense Advanced Research Projects Agency, or the United States Government. Reproduction in whole or in part is permitted for any purpose of the United States Government.

This document has been approved
for public release and sale; its
distribution is unlimited.

82 09 15 012

AD A120584

DTIC FILE COPY

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER ONR-N00014-77-C-0373	2. GOVT ACCESSION NO. A120584	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) DEVELOPMENT OF A TABLETOP MODEL FOR THE GENERATION OF AMORPHOUS/MICROCRYSTALLINE METAL POWDERS		5. TYPE OF REPORT & PERIOD COVERED Final Report 01 May 1978 to 31 May 1982
		6. PERFORMING ORG. REPORT NUMBER 82-1
7. AUTHOR(s) John F. Mahoney Zef Shanfield Julius Perel Bernard E. Kalensher Scott Taylor		8. CONTRACT OR GRANT NUMBER(s) N00014-77-C-0373
9. PERFORMING ORGANIZATION NAME AND ADDRESS Phrasor Scientific, Inc. 1536 Highland Avenue Duarte, California 91010		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Procuring Contracting Officer Office of Naval Research/Dept. of the Navy 800 Quincy St., Arlington, Virginia 22217		12. REPORT DATE JULY 1982
		13. NUMBER OF PAGES
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) DCASMA Van Nuys-Pasadena Office 3452 East Foothill Blvd. Pasadena, California 91107		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Reproduction of this report in whole or in part is permitted for any purpose of the United States Government.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Electrohydrodynamics Microcrystals Rapid Cooling Rates Amorphous Alloys Powder Metallurgy Deposition Microspheres Rapid Solidification Splat Cooling		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Electrohydrodynamics (EHD) was used as a basis for the generation and control of charged liquid droplets in order to produce micro-size powders and splat coatings. The EHD technique requires the use of intense electric fields to gen- erate liquid droplets directly from the liquid state. This is achieved by delivering a molten alloy to the tip of a fine capillary nozzle to which high voltage is applied. Resistance heater technology was developed, resulting in greater reliability and temperatures of 1800K were achieved. Powders smaller		

DD FORM 1 JAN 73 1473

EDITION OF 1 NOV 65 IS OBSOLETE III

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

than 0.01 micron have been produced with calculated cooling rates of approximately 10^7 K/s from radiation cooling alone. Several aluminum alloys were sprayed and produced small single spherical crystals, microcrystals, and adherent coatings. Amorphous powders and coatings (due to splat cooling) were formed using a Fe-Ni-B-P metallic glass alloy. Thin films were produced by EHD deposition of Si and Ge semiconductor materials. An Engineering Model was developed to study operation of an EHD powder generator using automation and modern computer techniques. Results of these studies were used to design, construct, and deliver a Micro-Particle Processor for use as a research instrument for experimentation with fine powders, and the production of coatings and thin films, involving rapid solidification.

The end product of the program, the Micro-Particle Processor was delivered to the Metallurgical Division of the National Bureau of Standards. Acceptance tests were performed and the Processor was used to run several new alloys. The manual for the Processor is provided as an appendix to this report.

FOREWORD

The information presented in this report was generated under ARPA Order Number 3363 and ONR Contract Number N00014-77-C-0373. The work was performed by Phrasor Scientific, Inc., Duarte, California.

This program was initiated under the direction of Dr. Edward C. van Rauth, then coordinated under Lt. Col. L. Jacobson, Materials Science, Defense Advanced Research Projects Agency. The Scientific Officer is Dr. Bruce A. McDonald, Materials Science Division, Office of Naval Research, Arlington, Virginia.

The principal investigator for the program was Mr. John F. Mahoney (213) 357-3201 of Phrasor Scientific, Inc., 1536 Highland Avenue, Duarte, California 91010. Work on the program was also performed by Dr. Julius Perel, Mr. Scott Taylor, Dr. Zef Shanfield, Dr. Bernard E. Kalensher, Mr. J. Robert Otto, and Mr. Paul Pauls. In addition, consultation assistance was obtained from Professor Pol Duwez, Dr. Robert Mehrabian, Dr. Carlos Levi, Dr. Jon Malvin, and Mr. Hoyt Todd.

The view and conclusions contained in this document are those of Phrasor Scientific and should not be interpreted as necessarily representing the official policies, either expressed or implied, of the Defense Advanced Research Projects Agency, Office of Naval Research or the United States Government.



Accession For	
NTIS	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
Distribution	
Availability	
Dist	A

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1. INTRODUCTION	
2. MICROSTRUCTURES OF POWDERS GENERATED BY THE MICRO-PARTICLE PROCESSOR	
2.1 Capabilities of the Micro-Particle Processor	
2.2 Microstructures of Aluminum Powders	
2.3 Segregation Behavior and Interface Stability	
2.4 Nucleation Behavior	
3. INSTALLATION OF THE MICRO-PARTICLE PROCESSOR AT THE NATIONAL BUREAU OF STANDARDS	
3.1 Shipping and Installation	
3.2 Initial Powder Processing	
3.3 Recommendations	

APPENDIX I. MICRO-PARTICLE PROCESSOR USERS' MANUAL

References

LIST OF FIGURES

PAGE

1. TEM image and diffraction pattern of an Al-6% Si EHD powder, showing the transition between plane front and cellular growth modes due to segregation
2. TEM image and diffraction pattern of a single crystal Al-3% Si EHD powder.
3. TEM images of a powder containing 3 grains with different crystalline orientations. The diffraction pattern corresponds to the largest grain (upper right hand corner)
4. Bright and Dark Field images of twin crystals in a pure aluminum powder. The diffraction pattern shows the perpendicular to the twin plane (111), and the twin direction (422)
5. Typical appearance of Al-Ge eutectic powders supported on a carbon film
6. TEM image and micro-diffraction patterns of an amorphous Al-Ge eutectic powder and the surrounding carbon film
7. TEM image and micro-diffraction pattern of an Al-Ge eutectic powder
8. Examples of bicrystals in Al-Ge eutectic powders
9. Typical structure of powders above 100nm in diameter of Al₃₀Ge
10. Submicron powder showing a large crystal of Al (A) and a zone with smaller crystals of a Ge-rich phase (B)

SECTION 1. INTRODUCTION

In May 1977, the ARPA initiated a program with Phrasor Scientific, Inc. to (1) develop the method of high temperature electrohydrodynamics (EHD), (2) apply this technology to rapid solidification and other materials-oriented areas, and (3) produce a unique laboratory instrument capable of generating micron and submicron particles of various alloy systems. Before the initiation of this contract little, if any, technical information existed in the field of high temperature EHD to draw upon the experience of previous research or state-of-the-art development. This program has succeeded in introducing into Materials Science a unique material processing and research tool based on advances made in the understanding and construction of molten metal EHD devices during the past few years. The integration of these two very distinct and separate technologies, EHD and Materials Science, has stimulated an interest in this program by various government laboratories, private industry, and universities. The nature of these outside inquiries (foreign and domestic) cover such diverse applications as rapid solidification, nucleation studies, magnetic properties of fine powders, powders composed of refractory metal and ceramic materials, rare earth and precious metal applications, copper powders for dispersion in inks used with hybrid circuits, photovoltaics, novel coatings and others.

As with many new technology developments, the transfer of new concepts to other fields has been rewarding. The capability of the EHD process to deposit thin films and coatings has been evaluated as a process that could produce low-cost polycrystalline silicon sheet for solar photovoltaic applications (Ref. 1). A new technology is now being explored for the purification of thin metallic foils and thin sheets comprised of semiconducting materials such as silicon and germanium (Ref. 2). Perhaps a lower cost method of producing detector grade silicon will emerge from this purification process.

The main end objective of this program was to develop an automated Micro-Particle Processor (μ PP), capable of producing rapidly solidified

powders in laboratory quantities for use by material scientists. This work was motivated by the possibility of processing new material structures and compositions by rapid solidification. Amorphous powders and new alloy compositions were originally anticipated and have been produced by EHD. Much more work in metallurgy is required to increase our understanding of the interrelationship between various process variables involved in rapid solidification. These variables, such as alloy composition, cooling rate, microstructure, etc. can only be obtained through careful experimentation and imaginative theoretical considerations. The Micro-Particle Processor is able to provide controlled and reproducible scientific experimentation affordable in a relatively small research laboratory. EHD opens new areas of investigation and could play a major role in studies of the rapid solidification for a wide variety of alloy compositions under controlled conditions. The μ PP provides a means for producing submicron droplets of both low and high temperature alloys which are rapidly quenched in flight or by deposition on various substrates (conductive cooling). It could become an important instrument for fundamental and applied studies of rapid solidification and segregation phenomena, in addition to the development of new alloy compositions that satisfy the material requirements of a rapidly expanding technological society.

Section 2 of this report discusses typical microstructures of rapidly solidified powders generated by the Micro-Particle Processor. The delivery, installation and operation of the μ PP successfully developed on this program is described in Section 3. Included in this final report (Appendix I) is the Micro-Particle Processor Users' Manual compiled for the National Bureau of Standards where the first Processor was delivered. The manual discusses in detail the μ PP system modules, preparation of the μ PP for powder processing, automated computer-controlled operation of the μ PP and basic system maintenance. For details of the technological development of high temperature electrohydrodynamics or liquid metal atomization, the reader is referred to a previously published Interim Technical Report given in Reference 3.

SECTION 2. MICROSTRUCTURES OF POWDERS GENERATED BY THE MICRO-PARTICLE PROCESSOR

2.1 Capabilities of the Micro-Particle Processor

The capabilities of the Micro-Particle Processor to produce rapidly quenched metallic powders for the materials scientist are listed below:

- a. Solidification studies relating average cooling rate, prior to solidification, and particle size to the formation of amorphous and crystalline structures.
- b. Rapid solidification studies performed to determine the effect of particle size and cooling rate on increases in solute solubility of alloyed elements.
- c. Rapid solidification and heat transfer studies on extremely fine crystalline and glass metal powders (Ref. 4).
- d. Production of electron transparent metal powders. The direct observation using TEM of a complete casting is a unique capability heretofore not available.
- e. Nucleation studies could be made that would relate particle size to the number of crystals (number of nuclei) in each particle.
- f. Electron diffraction study of fine powders to establish the sequence of formation of stable and metastable phases in a given alloy system as a function of cooling rate.
- g. The electron transparency of the fine powders produced permits studies of the growth morphology (plane front, cellular, or dendritic) during solidification. Effects of particle size, cooling rate, and solute content upon liquid-solid interface morphology can be studied.

Several of the above listed capabilities have been demonstrated (Ref. 5, 6,7,8).

2.2 Microstructures of Aluminum Powders

It is commonly accepted that the microstructural benefits produced by Rapid Solidification Processing (RSP) are determined by the

transport processes occurring at the liquid-solid interface, hence being dependent on the growth rate (i.e., local undercooling). In general, RSP effects may then be achieved either by rapid cooling during solidification and/or by substantially undercooling the liquid prior to nucleation. This latter approach is usually regarded as necessary to produce certain metastable structures like extended solid solutions or amorphous alloys (Ref. 9).

Several investigators (Ref. 10,11,12,13) have shown that emulsions of liquid metals and alloys can achieve high undercoolings prior to solidification because the active nucleation catalysts are isolated in a small fraction of the total volume. Their results indicate that the maximum undercooling -- which ranged from $0.26T_M$ for Pb to $0.5T_M$ for Ga (Ref. 13), is a function of the surface coating (emulsifier) and the droplet size distribution.

Vacuum atomization via EHD has proven itself a reliable tool for the controlled generation of powders below $1\mu\text{m}$ in diameter. These powders, while not yet of commercial importance, are especially suited for fundamental solidification studies for two reasons. First, the atomized liquid droplets are likely to undercool substantially due to their size and the minimization of surface films. Secondly, the powders are electron transparent, making it possible to analyze the microstructure of a complete "casting" without need for sectioning or thinning.

EHD submicron aluminum powders supplied by Phrasor Scientific, Inc. have been used for some time in solidification research at the University of Illinois (Ref. 4). Extensive studies were done on pure Al, Al-Si, Al-Cu, and Al-Ge alloys, and the results are briefly reviewed below.

2.3 Segregation Behavior and Interface Stability

Research in the Al-6% Si system revealed that submicron powders (especially those below 200nm) solidify in a plane-front mode without apparent segregation at Si contents as high as 6% (maximum equilibrium

solubility is 1.65%). This extended solubility indicates the achievement of large undercoolings prior to nucleation. In the larger particles, there was usually a transition from plane-front to cellular (segregated) growth as the droplet size increased and the achievable undercooling was consequently decreased. Fig. 1 shows an example of the interface breakdown due to segregation in an Al-6% Si powder.

2.4 Nucleation Behavior

Most of the submicron powders are expected to be monocrystalline (e.g., Fig. 2) because the growth velocities at high undercoolings are likely to produce solidification of the entire particle from the first nucleation event. However, it was found that a substantial proportion of the submicron powders are polycrystalline aggregates resulting from multiple nucleation (e.g., Fig. 3), and growth twins (e.g., Fig. 4). The analysis revealed that increasing undercooling by reducing the particle size promotes both of the above structures. Polycrystalline aggregates are a consequence of a sharp increase in the nucleation frequency at a certain level of undercooling, thereby making the nucleation competitive with the growth process. Twins are believed to originate mainly from stacking "accidents" during nucleation, producing a coherent boundary that propagates during growth.

Current investigations in EHD submicron powders are focused on eutectic alloys, where work is underway in $\text{Al}_{70}\text{Ge}_{30}$ and will soon be started in $\text{Al}_{88}\text{Si}_{12}$. The Al-Ge system features a simple eutectic at 30.3 at. %Ge and 697K. The equilibrium phases are fcc aluminum solid solution (2.8 at. %Ge max.), and diamond-cubic germanium (0.97 at. %Al max.). Rapid solidification has been reported to produce a variety of metastable phases, including amorphous regions in splats quenched on a diamond substrate (Ref. 14). The more recent investigations (Ref. 15) support the formation of an Al-rich rhombohedral phase at ~ 6 at. %Ge, and a Ge-rich monoclinic phase at ~ 60 at. %Ge.

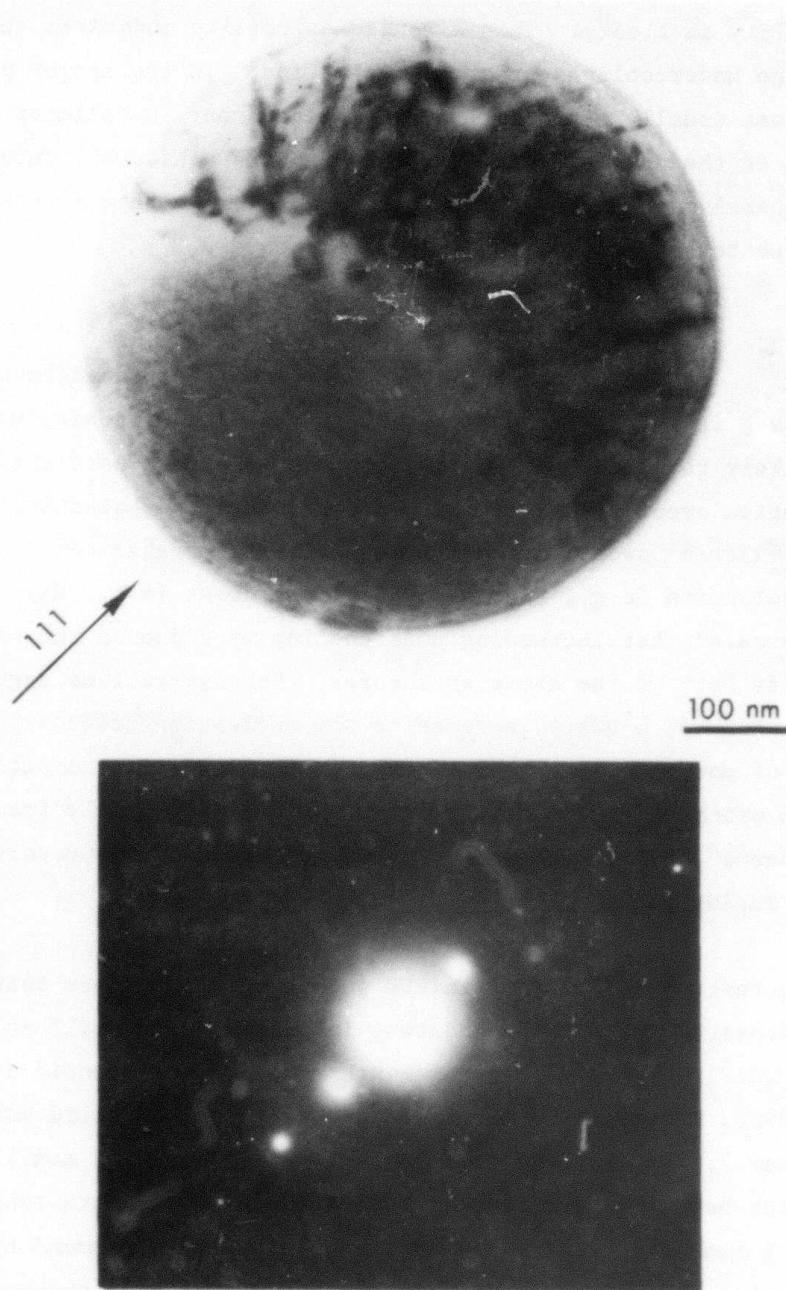


FIGURE 1.

TEM image and diffraction pattern of an Al-6%Si EHD powder, showing the transition between plane front and cellular growth modes due to segregation.

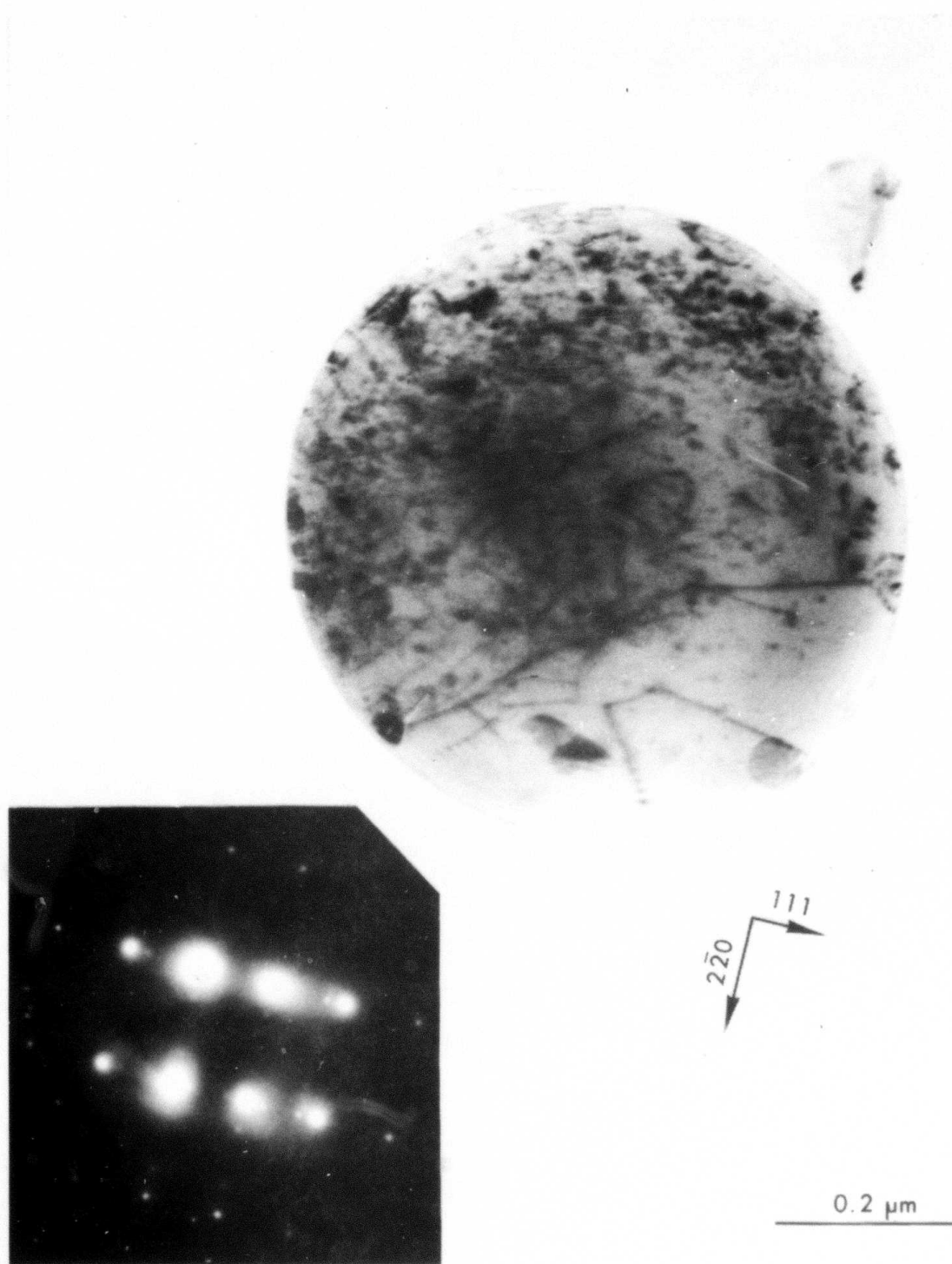
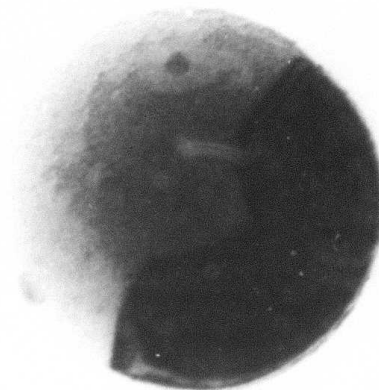
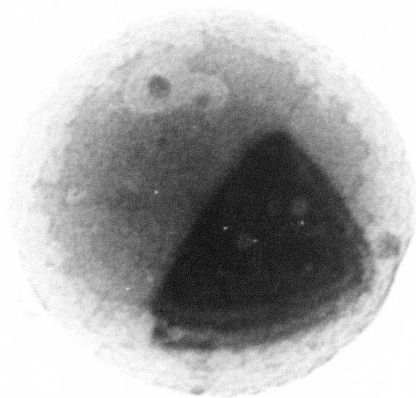
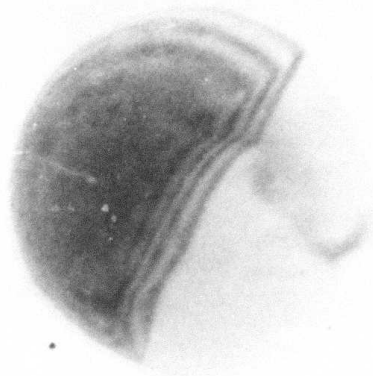
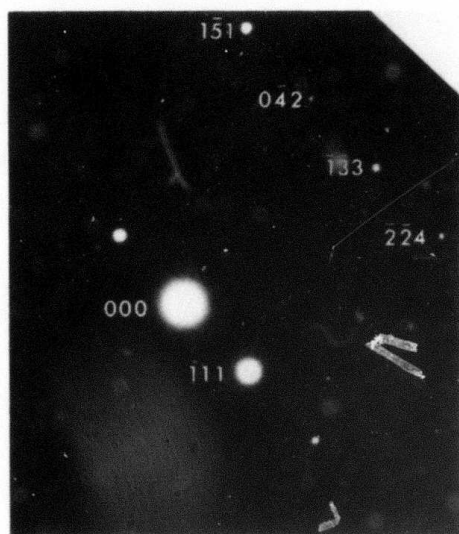


FIGURE 2.

TEM image and diffraction pattern of a single crystal Al-3%Si EHD powder.



0.1 μm

FIGURE 3.

TEM images of a powder containing 3 grains with different crystalline orientations. The diffraction pattern corresponds to the largest grain (upper right hand corner). (Al-4.5% Cu)

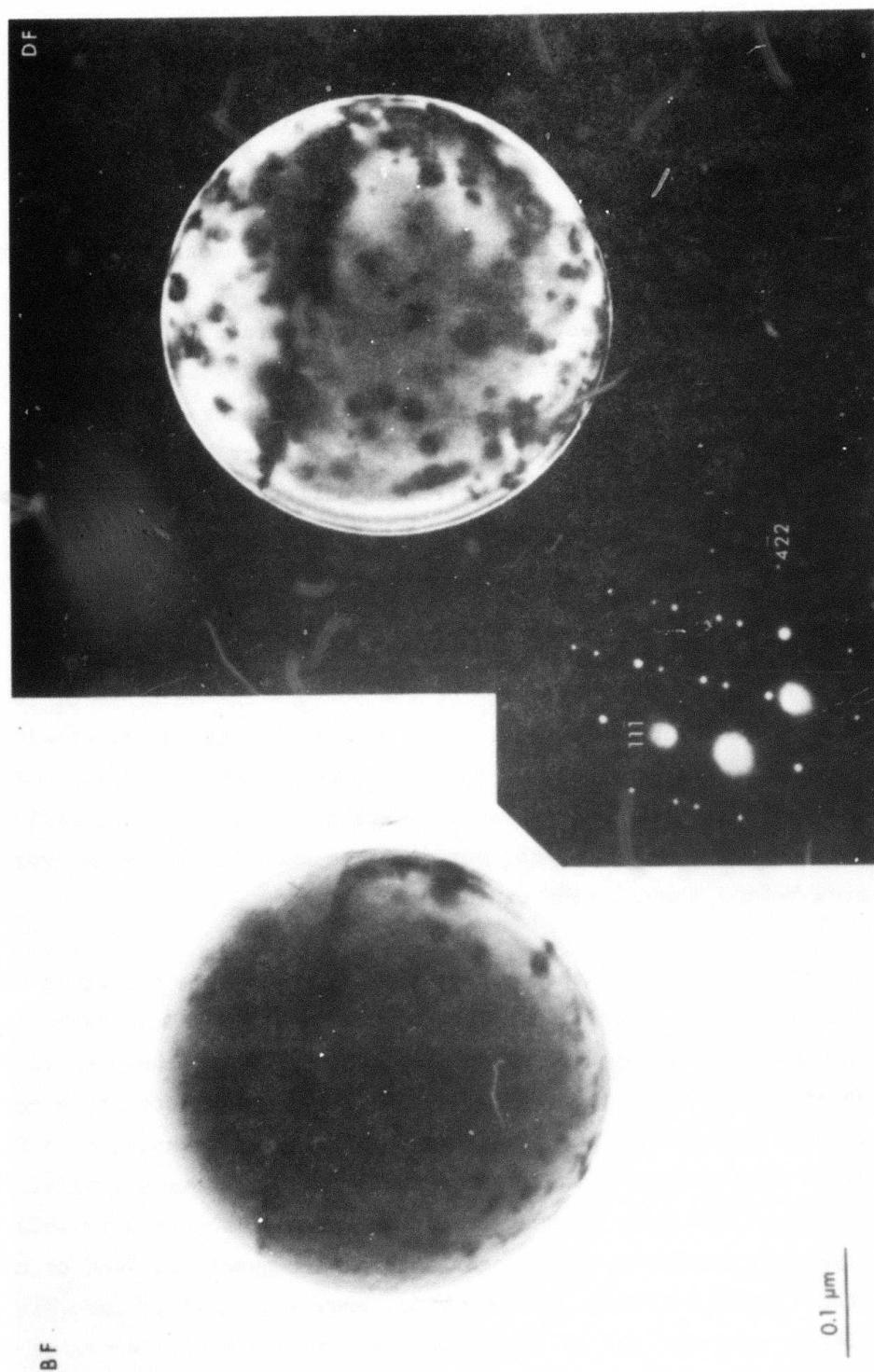


FIGURE 4.

Bright and Dark Field images of twin crystals in a pure aluminum powder. The diffraction pattern shows the perpendicular to the twin plane ($11\bar{1}$) and the twin direction $[4\bar{2}2]$.

Preliminary work in Al-Ge eutectic powders involves the identification of typical microstructures present in the submicron range. A typical specimen is shown in Fig. 5. Results up to date are briefly described below.

a) A small fraction of the powders, usually below 100nm in diameter, exhibit an amorphous structure such as that in Fig. 6. Microdiffraction techniques, which enable information to be derived from an area equivalent or smaller than the particle, revealed distinct sets of amorphous rings for the powder and the supporting carbon film. When the same technique is applied to a crystalline particle of comparable size, as in Fig. 7, there is a clear set of diffraction spots. We may then conclude that the powder in Fig. 6 is completely amorphous and was, therefore undercooled below the glass transition temperature before nucleation could occur.

b) A large fraction of the powders in the range of 200nm or smaller are bicrystals (e.g., Fig. 8) although it is not clear at the present time if they are of the same phase. Some preliminary diffraction and microchemical information suggests that one of the crystals may be Al-rich and other, Ge-rich, but there is need for extensive work in this area before a conclusion can be reached.

c) Most of the powders above 100nm have a structure similar to that in Fig. 9, where a large number of crystals in the range of 10nm can be distinguished by diffraction contrast. Some of the powders that could be properly tilted (e.g., Fig. 10) revealed a microstructure reminiscent of that produced by the plane-front to cellular transition in Fig. 1. Micro diffraction of the light area in Fig. 10 shows a typical fcc Al pattern (110 zone), whereas the darker areas (in contrast) show a polycrystalline pattern with larger interplanar spacings, such as expected from a Ge-rich phase. The evidence, however, is still insufficient to clearly identify all of the phases present and hence determine whether

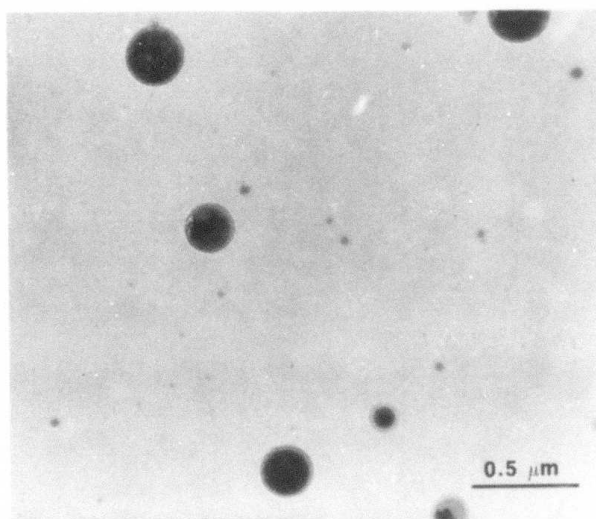
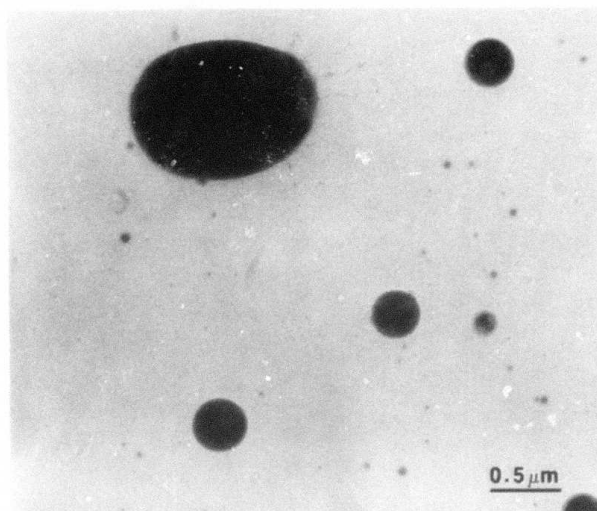
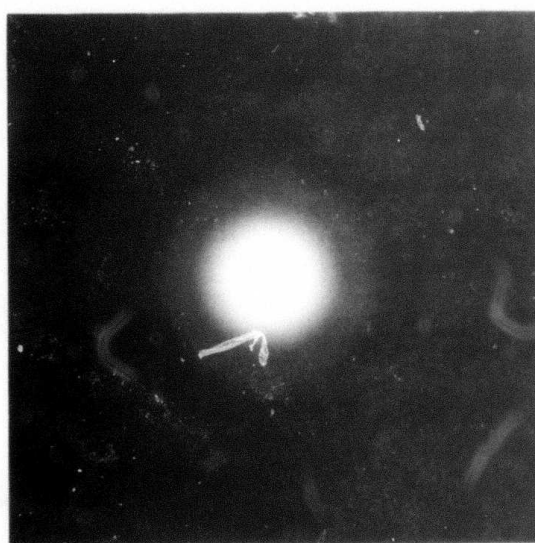
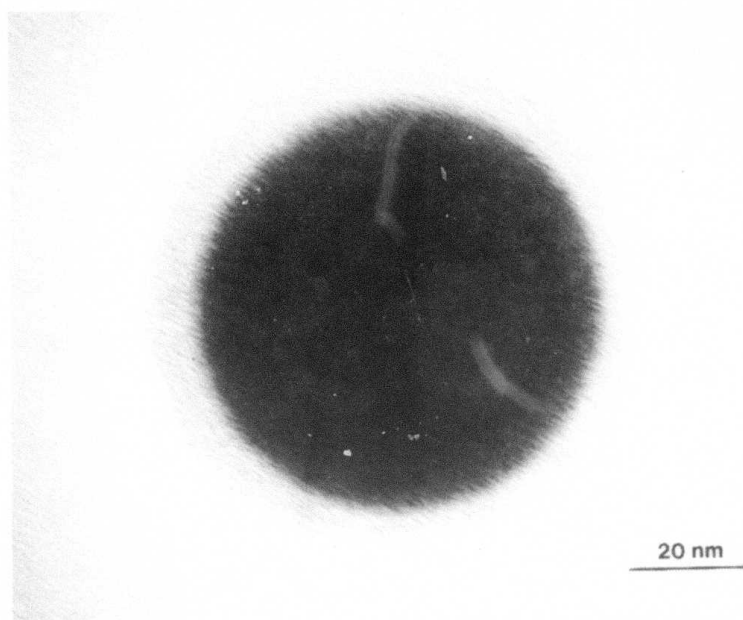
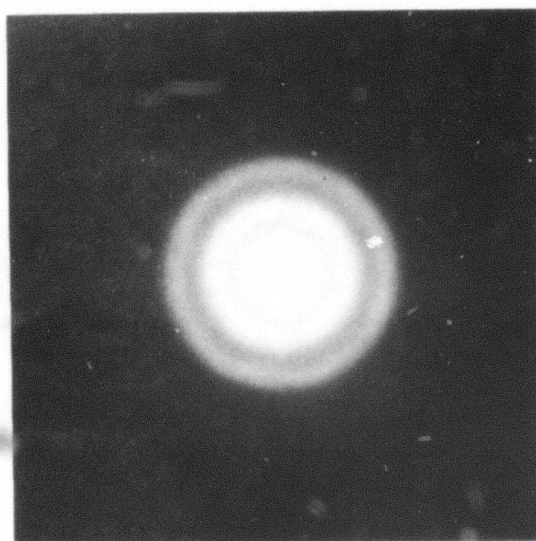


FIGURE 5. Typical appearance of Al-Ge eutectic powders supported on a carbon film.



CARBON FILM



POWDER

FIGURE 6. TEM image and micro-diffraction patterns of an amorphous Al-Ge eutectic powder and the surrounding carbon film.

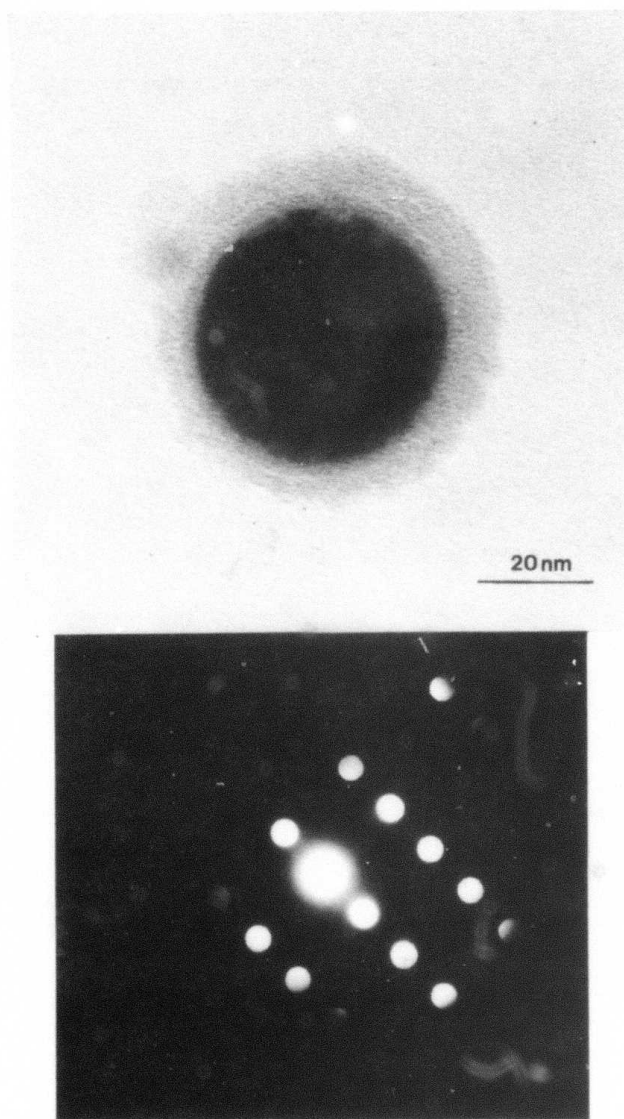
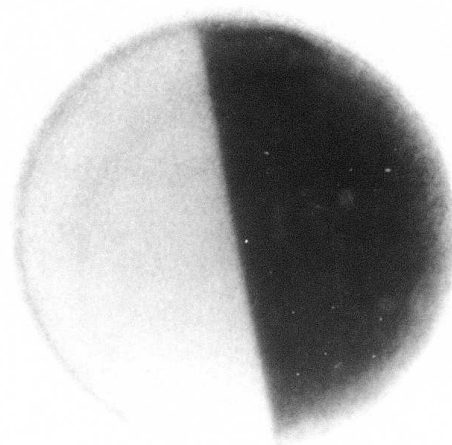


FIGURE 7.

TEM image and micro-diffraction pattern of an Al-Ge eutectic powder.



50 nm



50 nm

FIGURE 8. Examples of bicrystals in Al-Ge eutectic powders.



50 nm

FIGURE 9.

Typical structure of powders above 100 nm in diameter
of Al-30% Ge.

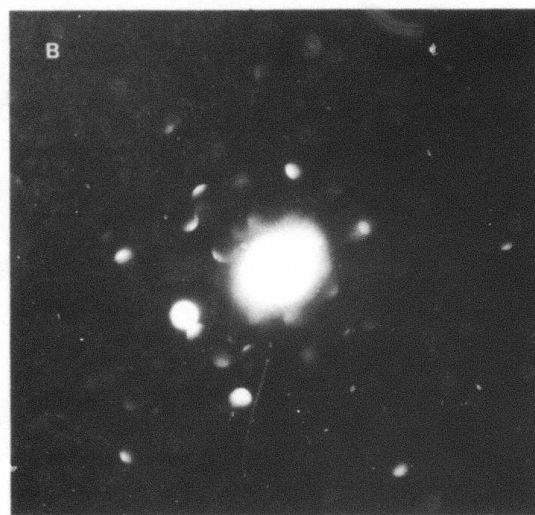
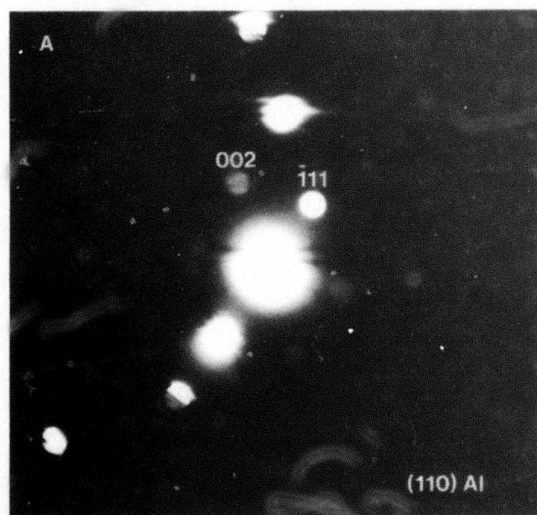
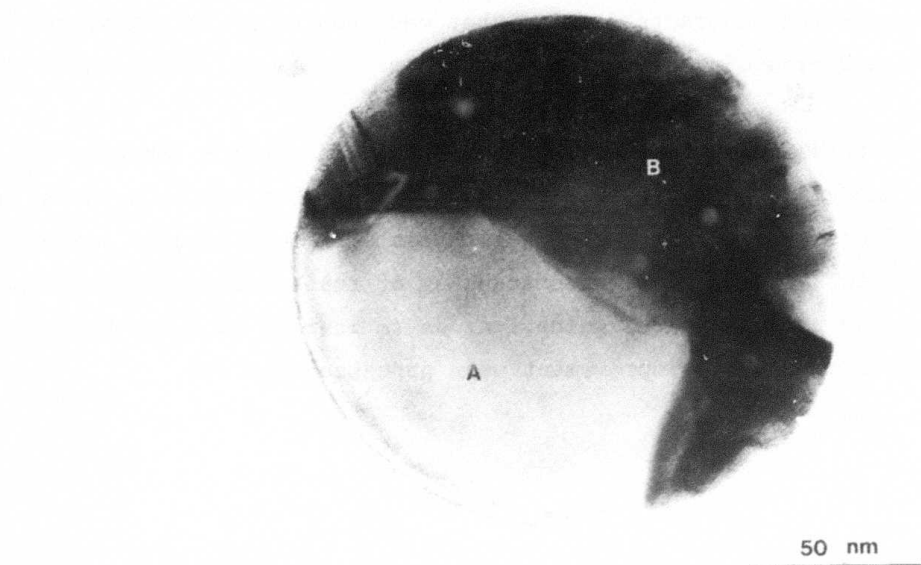


FIGURE 10.

Submicron powder showing a large crystal of Al (a) and a zone with smaller crystals of a Ge-rich phase (B).

these are metastable structures produced by undercooled solidification.

Further work in the Al-Ge system should involve phase identification and microchemical characterization that can eventually be translated into a better understanding of the solidification mechanisms in these alloys.

It is becoming increasingly evident that EHD powders can be a valuable tool in studying the achievable microstructures that can be produced by rapid solidification in a certain alloy system. Such studies are not only necessary for a fundamental understanding of the solidification phenomena, but may also provide useful guidelines as to whether an alloy has a good potential for property improvement when processed via RSP.

SECTION 3. INSTALLATION OF MICRO-PARTICLE PROCESSOR AT THE NATIONAL BUREAU OF STANDARDS

3.1 Shipping and Installation

The Micro-Particle Processor was shipped from Phrasor Scientific, Inc. laboratory by air freight on 11 May 1982. The system arrived in excellent condition at NBS on 12 May 1982. Scott Taylor of Phrasor Scientific, Inc. arrived at NBS on 14 May to aid in the inspection, installation and initial operation of the μ PP system. Key personnel at NBS who assisted in the preparation and operation of the μ PP included Dr. Steve Ridder and Mr. Frank Biancaniello.

3.2 Initial Powder Processing

After solving some initial problems related to computer halting and high voltage power supply signals, the system was ready for processing rapidly solidified powders. On 16 May, a processing run was made using an Al-Mn(9%) alloy. After the run, the processing chamber was cleaned and the Micro-Particle Source was loaded with an Al-Mn(3%) alloy to demonstrate μ PP performance for visitors from the Department of Commerce on 17 May. The demonstration was successfully completed, and the powder samples were removed for electron microscope analysis. Later, on the 17th of May, an Al-Fe(34%) alloy was successfully processed.

3.3 Recommendations

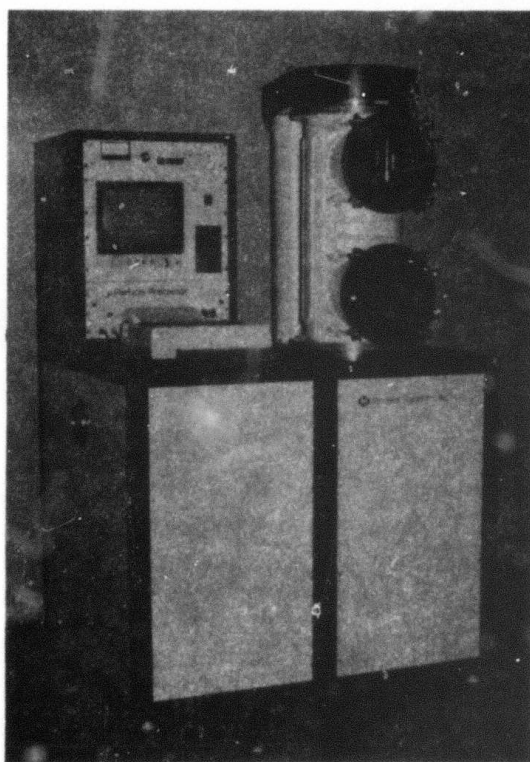
Various suggestions were put forth by personnel at NBS to improve the performance of the Micro-Particle Processor. It was recommended that the system be modified to vent the chamber with an inert gas while the source is still hot to decrease the cool-down cycle time. This would increase the number of sample runs that could be made in a single day. This modification could double the number of powder runs from three to six in a typical working day. It was also suggested that a second rough pump be used to evacuate the feedpressure system. This would allow a faster feedpressure response time without affecting the foreline pressure.

REFERENCES

1. J.F.Mahoney and J.Perel, "Electrostatic Spray of Silicon For Photovoltaic Applications", IEEE/IAS 1981 Annual Meeting, Philadelphia, PA (5-9 October 1981).
2. J.Perel and J.F.Mahoney, "High Field Purification of Silicon", 160th Meeting Electrochemical Society, Inc., Symposium on Silicon Purification, Denver, CO, Extended Abstracts, p.1152 (11-16 October 1981).
3. J.F.Mahoney, J.Perel, Z.Shanfield and B.E.Kalensher, "Development of a Tabletop Model for the Generation of Amorphous/Microcrystalline Metal Powders", Interim Technical Report #80-1, Office of Naval Research, Arlington, VA (July 1980).
4. C.G.Levi and R.Mehrabian, "Microstructures of Rapidly Solidified Aluminum Alloy Submicron Powders", Metallurgical Trans., 13A, 13 (1982).
5. J.Perel, J.F.Mahoney, B.E.Kalensher, K.E.Vickers and R.Mehrabian, Rapid Solidification Processing: Principles and Technologies, edited by R.Mehrabian, B.H.Kear, M.Cohen (Claitor's Publishing Division, Baton Rouge, LA, 1978) p.258.
6. J.Perel, J.F.Mahoney, B.E.Kalensher and R.Mehrabian, "Electrohydrodynamic Techniques in Metal Processing", Twenty-fifth Sagamore Army Materials Conference on: Recent Advances in Metal Processing, Bolton Landing, New York, (17-21 July 1978).
7. J.Perel, J.F.Mahoney, Z Shanfield, S.Taylor and C.Levi, "An Automated Laboratory Apparatus for the Production of Rapidly Solidified Sub-Micron Powders", The Conference on Rapidly Solidified Metastable Materials, Boston, MA (17-19 November 1981).
8. J.F.Mahoney, "Tiny Particles Aid Research on Microstructures", Industrial Research & Development, 24, 150 (January 1982).
9. M.Cohen, B.H.Kear and R.Mehrabian, Rapid Solidification Processing: Principles and Technologies II, Proceedings of the Second International Conference on Rapid Solidification Processing in Reston, VA on 23-26 March 1980. R.Mehrabian, B.H.Kear and M.Cohen, Editors, Baton Rouge, LA, Claitor Publishing Division, 1980.
10. D.Turnbull, J.Chem.Phys. 20, 411 (1952).
11. J.H.Hollomon and D.Turnbull, Probl Metal Phys., 4, 333 (1953).

12. J.H.Perepezko, D.H.Rasmussen, I.E.Anderson and C.R.Loper, Jr., Solidification and Casting of Metals, Proceeding of a Conference at the University of Sheffield, July 18-21, 1977. London: The Metals Society, 1979, p.169.
13. J.H.Perepezko, Rapid Solidification Processing: Principles and Technologies II, Proceedings of the Second International Conference on Rapid Solidification Processing in Reston, VA on March 23-26, 1980. R.Mehrabian, B.H.Kear and M.Cohen, Editors, Baton Rouge, LA, Claitor Publishing Division, 1980.
14. P.Ramachandrarao, M.Laridjani and R.W.Cahn, Z.Metallkunde 63, 43 (1972).
15. M.Laridjani, K.D.Krishnanand and R.W.Cahn, J. Mater, Science 11, 1643 (1976).

MICRO-PARTICLE PROCESSOR USERS MANUAL



 **Phrasor Scientific, Inc.**

1536 HIGHLAND AVENUE
DUARTE, CALIFORNIA 91010

TABLE OF CONTENTS

	<u>PAGE</u>
List of Figures	iii
List of Tables	v
1. GENERAL INFORMATION	1-1
1.1 Introduction	1-1
1.2 Utility Requirements	1-2
1.3 Specifications	1-3
1.4 Major Equipment Manuals	1-5
2. SYSTEM DESCRIPTION	2-1
2.1 Introduction	2-1
2.2 System Modules	2-6
2.3 Description of μ PP Printed Circuit Boards	2-7
2.4 Micro-Particle Source	2-8
2.5 Tape Drive Assembly	2-13
2.6 Vacuum System and Pump Stack	2-13
2.7 Feedpressure Manifold	2-18
2.8 High Voltage Isolation Module	2-18
2.9 Relay Module	2-22
2.10 Suppression of Electromagnetic Noise	2-22
2.11 Source Temperature Measurement and Control System	2-33
2.12 Main Console Connections	2-38
3. PREPARATION FOR POWDER PROCESSING	3-1
3.1 Unloading Reservoir Assembly from Micro-Particle Source	3-1
3.2 Installing Reservoir Assembly Into Micro-Particle Source	3-3
3.3 Loading the Tape Drive Assembly	3-5
3.4 Unloading the Tape Drive Assembly	3-10

TABLE OF CONTENTS Continued

	<u>PAGE</u>
4. OPERATION OF THE MICRO-PARTICLE PROCESSOR	4-1
4.1 General Directions	4-1
4.2 Turning on uPP	4-1
4.3 Standby Mode	4-2
4.4 Parameter Setup Mode	4-5
4.5 Startup Vacuum Pumps	4-7
4.6 Chamber Pumpdown Mode	4-10
4.7 Source Heatup and Operation	4-14
4.8 Shutdown Mode	4-22
5. MAINTENANCE	5-1
5.1 Removal of Top Flange from Vacuum Chamber	5-1
5.2 Disassembly of Micro-Particle Source	5-7
5.3 Assembly of Micro-Particle Source	5-12
5.4 Removal of Tape Drive Assembly from Vacuum Chamber	5-17
5.5 Nozzle Heater Construction	5-19
5.6 Reservoir Heater Construction	5-19
5.7 Micro-Particle Source Maintenance Kit	5-22

LIST OF FIGURES

<u>FIGURE</u>		<u>PAGE</u>
2-1	Micro-Particle Processor	2-2
2-2	Micro-Particle Processing Chamber	2-3
2-3	μPP Functional Block Diagram	2-5
2-4	Printed Circuit Boards	2-9
2-5	Printed Circuit Boards	2-10
2-6	Micro-Particle Source	2-11
2-7	Tape Drive Base Plate Components	2-14
2-8	Tape Drive Stepper Motor	2-15
2-9	Tape Drive Shutter Solenoid	2-16
2-10	Vacuum System and Pump Stack	2-17
2-11	Reservoir Feed System Module	2-19
2-12	Reservoir Feed System Valve Schematic	2-20
2-13	High Voltage Isolation Physical Layout	2-21
2-14	High Voltage Isolation Module Connections	2-23
2-15	Relay Module Panels Showing Connectors	2-24
2-16	Relay Module Panel (Rear)	2-25
2-17	Relay Module Panel (Left Side)	2-26
2-18	L-C Filter Assembly	2-34
2-19	High Voltage Isolation Module L-C Filter Assembly	2-35
2-20	Block Diagram of Thermocouple Isolation Circuitry	2-36
2-21	Main Cabinet Connections (Rear)	2-39
3-1	Gas Inlet and Reservoir Clamp Components	3-2
3-2	Ceramic Reservoir Assembly	3-4
3-3	Tape Drive Assembly	3-7
3-4	Tape Drive Assembly Schematic	3-8

LIST OF FIGURES Continued

<u>FIGURE</u>		<u>PAGE</u>
4-1	Video Screen Displays (Standby and Parameter Setup Modes)	4-3
4-2	Video Screen Displays (Startup Vacuum and Chamber Pumpdown Modes)	4-8
4-3	Video Screen Displays (Source Heatup and Shutdown Mode)	4-15
5-1	Micro-Particle Source as Seen Through Door of Cylindrical Heat Shield	5-2
5-2	Top and Base Plate Feedthrough Locations	5-4
5-3	EMI Shield Box for Source Flange Insulators	5-6
5-4	Standoffs and Supports Referred to in Micro-Particle Source Assembly and Disassembly Instructions	5-8
5-5	Location of Source Parts Referred to in Disassembly Instructions	5-10
5-6	Reservoir Heater Lead Assembly	5-11
5-7	Disassembled Configuration of Micro-Particle Source	5-13
5-8	Flexible Coupling Assembly	5-18
5-9	Nozzle Heater Construction	5-20
5-10	Reservoir Heater Construction	5-21
5-11	Micro-Particle Source Maintenance Kit	5-23

LIST OF TABLES

<u>TABLE No.</u>		<u>PAGE</u>
1-1	Utility Requirements	1-2
1-2	List of Major Equipment Installed on Micro-Particle Processor	1-5
1-3	Electronic Circuit Schematics and Block Diagrams Supplied with Basic μPP Users' Manual	1-6
2-1	System Modules	2-6
2-2	Printed Circuit Board Descriptions	2-7
2-3	Description of Source Parts	2-12
2-4	High Voltage Isolation Module Physical Layout	2-18
2-5	Relay Module Panel Connector Designa- tions (Rear)	2-27
2-6	Relay Module Panel Connector Designa- tions (Left Side)	2-29
2-7	Relay Description	2-32
2-8	EMI Suppression Components	2-22
3-1	Tape Drive Assembly	3-9
5-1	Top and Base Plate Feedthrough Designations	5-5

SECTION 1

GENERAL INFORMATION

1.1 INTRODUCTION

This manual provides information for the operation and maintenance of the Phrasor Scientific, Inc. Micro-Particle Processor (μ PP) System. The Micro-Particle Processor is a laboratory instrument designed for rapid solidification processing of micron and submicron powders by radiative cooling. The μ PP can also produce rapidly solidified deposits (splats) on various substrates. The process, which is computer controlled, produces liquid metal droplets formed by electrohydrodynamics (EHD) atomization in a vacuum environment. For details on the theory and description of EHD droplet production, the user is referred to the documentation sources listed at the end of this manual. The μ PP system allows the material-oriented scientist to prepare rapidly solidified samples for the following research studies:

Metallic Glasses	New Alloy Composition
Extension of Solid Solubility	Grain Size
Nucleation	Chemical Homogeneity
Metastable Structures	Undercooling Effects

In addition to describing utility, system and droplet source specifications, this manual will inform the user on how to process and collect rapidly solidified samples. It also describes in detail the assembly and maintenance of the high temperature droplet source. A set of drawings and schematics supplied with this manual will provide the user with other detailed information concerning the operational electronics controlling various μ PP module functions. Some of the major components of the μ PP have their own manuals which are either supplied or can be obtained from the manufacturer.

1.2 UTILITY REQUIREMENTS

TABLE 1-1
UTILITY REQUIREMENTS

Electrical Power	120V, 15A, Single Phase, 60 Cycle: Four Separately Fused Circuits Required
Cooling Water	15 Gal/Hr. for Diffusion Pump; the water must be clean, filter recommended. Cooling tubes are provided for the vacuum chamber and can be connected in series with the diffusion pump lines
Compressed Air	65 PSI, filtered, for gate valve activation
Argon Gas	10-20 PSIG regulator gauge; a controlled supply of Argon Gas required for reservoir pressurization
Liquid Nitrogen	A controlled supply of LN is required for the cryobaffle. A single baffle filling should last for 17 hours before a refill is necessary

1.3 SPECIFICATIONS

SOURCE:

Voltage.....1 to 20kV
Current.....0 to 3ma
Nozzle Heater Power.....500W Max.
Reservoir Heater Power.....500W Max.
Operating Temperature.....To 1350°C (2462°F)
Reservoir Pressure.....To 2000 Torr Max.
Reservoir Dimensions.....0.5" DIA x 1.5" high
(1.27 x 3.81cm)
Reservoir Volume.....0.3cm³ (4.8cm³)
Nozzle Orifice......002" DIA (.005cm)
Extractor Aperture.....3/16" DIA (0.476cm)

MATERIALS:

Reservoir.....High Purity Alumina (99.5%)
Nozzle.....High Purity Alumina (99.5%)
Source Gasket.....GRAFOIL (graphite)⁺
Extractor......030" THK Graphite ⁺⁺
Nozzle Heater......020" DIA Ta DR9^{*} wire 24" long
Reservoir Heater......020" DIA Ta DR9^{*} wire 60" long
Thermocouple.....Type K (Alumel-Chromel)

CONSOLES:

Main Console.....40" high by 47" wide by 40" deep
(101 x 119 x 101cm)
Computer Console.....27" high by 21" wide by 21" deep
(68 x 53 x 53cm)
Vacuum Chamber.....12" inside DIA x 27.5" high
(30 x 70cm)

+ Available from Union Carbide Corporation, Chicago, IL

++ Available from Poco Graphite, Decatur, TX

* Available from Kawecky Beryl Co., Boyertown, PA

CHARGE (TYPICAL):

Pure Elements.....Sn;Pb;Ge;Al;Cu;Au

Binary Alloys.....Pb-60%Sn;Al-4.5%Cu;Al-3%Si;
Al-6%Si;Al-12%Si;Al-25.2Fe;
Al-28%Cu;Al-1%Mn;Al-30%Ge

Amorphous Alloys.....Fe-15%Si-10%B;Fe-40%Ni-14%P-6%B;
Al-30%Ge

PARTICLE CHARACTERISTICS:

Size Range.....From less than .01 μ to 100 μ

Cooling Rates.....10⁴ to 10⁶k/sec

1.4 MAJOR EQUIPMENT MANUALS

Table I below lists the major equipment components of the μ PP. Those components have their own manual and are supplied with the basic Users Manual are indicated by the "checks". Other manuals can be obtained from the equipment manufacturer.

TABLE 1-2
LIST OF MAJOR EQUIPMENT INSTALLED ON MICRO-PARTICLE PROCESSOR

MANUFACTURER	EQUIPMENT	MODEL NO.	SERIAL NO.	MANUAL PROVIDED
Datametrics	Barocel Pressure Transducer	590-A-2000T 2P1-V1X-4D	103-8804	✓
Epson	Dot Matrix Terminal Printer	MX-80	356063	✓
RCA	Data Terminal Keyboard	VP3301	001193-3131	✓
RCA	Video Monitor	TC1212	o45170	*
Spellman	High Voltage Supply	RHR20P120	SP16506-1	✓
Tandon	Mini-Floppy Disc Drive	TM100-1	81114831	*
Andromeda	Turnkey Computer System	11A	216	*
Varian	Ionization Gauge Control	843	B329	✓
Varian	Roughing Pump	EVAC300	0402-K6810-701	✓
Airco Temescal	Pneumatic Vacuum Gate Valve	6"-5030R	Q1	*
Varian	Diffusion Pump	M-6	B079A	✓
Varian	Cryo-Baffle	0362-6	---	✓

* Contact Manufacturer

TABLE 1-3.
ELECTRONIC CIRCUIT SCHEMATICS AND BLOCK DIAGRAMS
SUPPLIED WITH BASIC MICRO-PARTICLE PROCESSOR USERS MANUAL

Circuit Schematics			
<u>Title</u>	<u>Drawing No.</u>	<u>P.C. Board Assembly No.</u>	<u>P.C. Board Location</u>
High Voltage Thermocouple Board and 20kV Isolation Transformer	06-005-01001	06-01-002A-01	H.V. Isolation Module
Low Voltage Thermocouple Board and Heater Controller	06-005-01002	06-01-001A-01	Computer
Feedpressure Controller	06-005-01003	06-01-001A-01	Computer
Stepper Motor Controller	06-005-01004	06-01-001A-01	Computer
Tape Drive Oscillator Freq. Counter	06-005-01005	06-01-001A-01	Computer
Vacuum Gauge Setpoint Board	06-005-01011	06-01-006A-01	Vacuum Gauge Controller
Quad Board Misc. Circuitry	06-005-01012	06-01-005A-01	Computer
Relay Module Circuit Board (includes stepper motor, shutter solenoid and feedpressure solenoids)	06-005-01013	06-01-005A-01	Relay Module
Source Current Board	06-005-01014	06-01-003A-01	H.V. Power Supply

Block Diagrams	
Temperature Controller	06-005-01006
Stepper Motor Controller	06-005-01007
Feedpressure Controller	06-005-01008
Tape Drive Oscillator Freq. Counter	06-005-01009

SECTION 2. SYSTEM DESCRIPTION

2.1 INTRODUCTION

The Micro-Particle Processor is shown in Fig. 2-1 and consists of the following major parts:

Computer Console:

Keyboard
Printer
Video Monitor
Main Switch
(Emergency Shutoff)
Floppy Disc Drive
Computer
Vacuum Gauge

Main Console:

H.V. Safety Cover
H.V. Tower
H.V. Module
H.V. Power Supply
Low Voltage Module
Relay Module

Vacuum System:

Chamber
Spool Piece
Gate Valve
Cryo-Baffle
Diffusion Pump
Roughing or Fore Pump
Foreline Valve
Roughing Valve
Vent Valve
Chamber and Foreline Thermocouple
(Gauges)

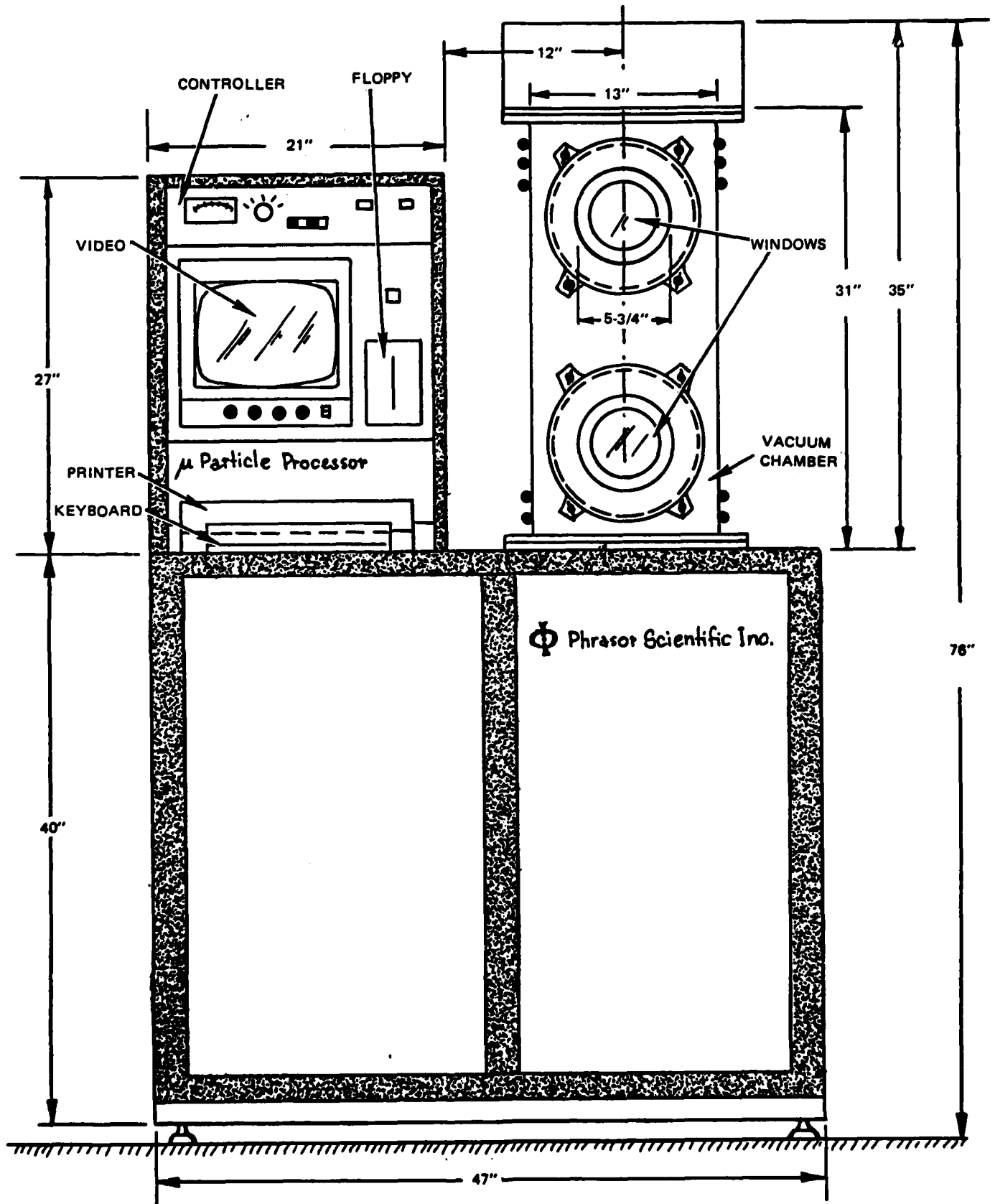
Feedpressure System:

Vacuum Outlet Solenoid Valve
Feedpressure Valve
Gas Inlet Solenoid Valve
Pressure Transducer

Fig. 2-2 depicts the vacuum system with the Micro-Particle source in operation and illustrates the basic function of the Processor: the generation of micron and submicron particle beams for subsequent collection on replicating tape mounted in a motorized tape drive assembly.

The Micro-Particle Processor requires a number of systems to support and control the electrohydrodynamic (EHD) source for generating sub-micron powders from molten alloy charges placed in the ceramic reservoir assembly. The various systems include a vacuum system, heater and high voltage power supplies, feedpressure manifold, particle collection system and a computer to monitor and control the particle generating process and

MICRO-PARTICLE PROCESSOR



(PROCESSOR IS 43 INCHES DEEP)

Fig. 2-1

TO HIGH VOLTAGE
ISOLATION MODULE

SAFETY COVER

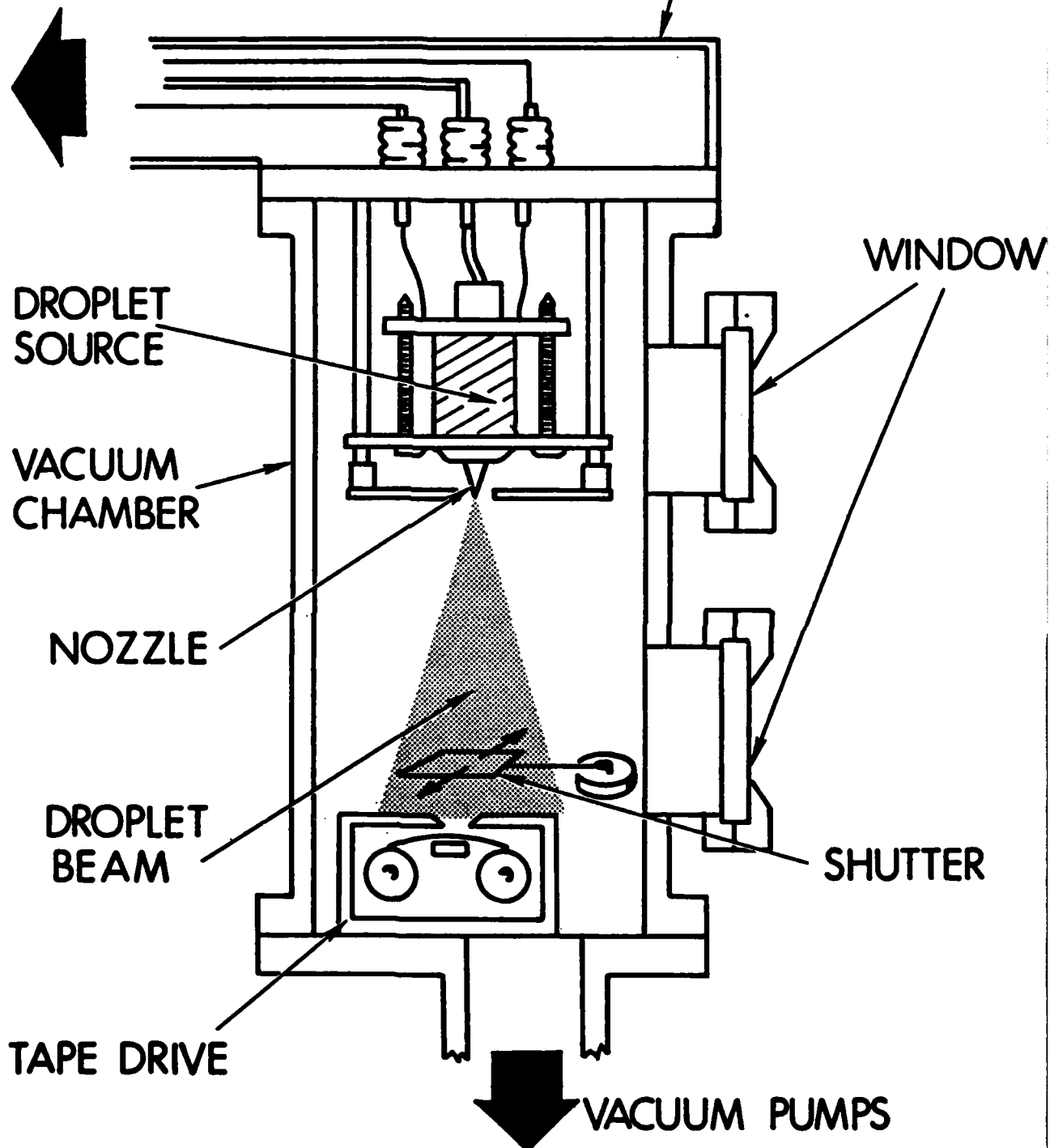


Fig. 2-2 Micro-Particle Processing Chamber

and communicate with the operator. These and other systems have been organized into a number of functional modules displayed in Fig. 2-3.

All operational controls are made through the keyboard shown at the top of the diagram in Fig. 2-3. The keyboard commands the computer which then communicates back to the operator via the video monitor (CRT). Questions are posed by the computer (software) and the status of various components is displayed on the CRT. The operator is able to make selections in relation to the questions and choose parameter values. Operational parameters such as temperatures, source voltage, feedpressure and vacuum chamber pressure are automatically printed when a sufficient change in value occurs or when the operator changes controllable independent parameters, such as source voltage, temperature and feedpressure. On the functional diagram, the droplet source is heated via the heater control (circuit) to set a temperature. The measured temperature is fed back to the computer and displayed on the CRT. Both the heater control and the high voltage are fed into the high voltage isolation module to enable the source to be heated while high voltage is applied. High voltage wires, which conduct heater current and thermocouple signals, extend from the isolation module through the high voltage tower located behind the vacuum chamber, and through the H.V. cover between the tower and top of the vacuum chamber. They then connect to the source through vacuum feedthroughs. EMI shielding is included in the Processor to eliminate noise interaction with computer electronics and other instruments. The CRT initially displays the status of all the vacuum system valves, the pressures, and a list of experimental modes. Including the initial one, the modes of operation are: a) Standby, b) Set parameter, c) Start vacuum pumps, d) Pumpdown chamber, e) Heat up and operation, f) Shutdown mode. By proceeding through this sequence, the operator is guided through the entire experiment by the computer which is designed to minimize operator errors.

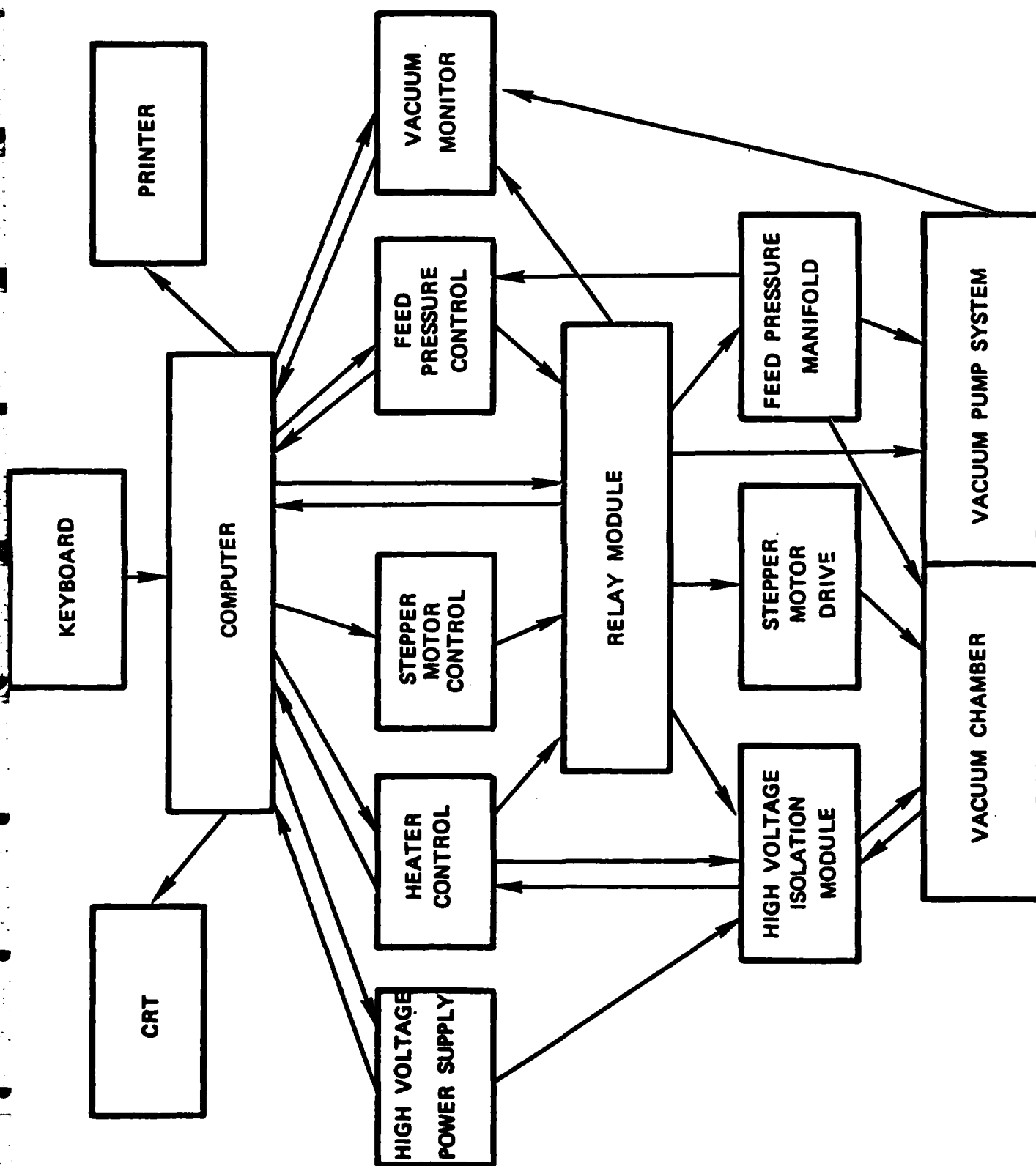


Fig. 2-3 uPP FUNCTIONAL BLOCK DIAGRAM

2.2 SYSTEM MODULES

Table 2-1 below lists the basic Micro-Particle system modules and a corresponding description of the module functions. Each module can be separately isolated for removal, replacement, modification or maintenance.

TABLE 2-1
SYSTEM MODULES

<u>MODULE</u>	<u>DESCRIPTION</u>
Source	The EHD source is used to generate particles by heating reservoir material above its melting point, delivering the molten metal to the nozzle by feedpressure or surface tension and extracting submicron droplets by application of high voltage.
Tape Drive Assembly	Tape drive with stepper motor is used to move replicating tape under a sample aperture for collection of particles. A shutter is mounted above the aperture. These are mounted within the vacuum chamber.
Vacuum Chamber	An enclosure in which molten particles are generated. Pressure during generation is 10^{-5} torr or better. The source and tape drive assembly are mounted within the vacuum chamber.
Pump Stack	Diffusion and roughing pumps, pneumatic valves and pressure gauges.
Vacuum Gauge Controller	Measures outputs of ion gauge and two thermocouple vacuum gauges on vacuum chamber and pump stack and triggers setpoint for computer intervention during pumping process.
High Voltage Isolation (HV-ISO) Module	Isolation and stepdown transformer for heater power, heater thermocouples, temperature measurement electronics and high voltage optoisolators, and solid state heater power control relays and optoisolators.

High Voltage Power Supply and Controller	Delivers 0-20kV of high voltage to the HV-ISO module as set by the computer.
Feedpressure Manifold	Plumbing, valves and pressure transducer (0-2000 torr) to supply feedpressure to deliver molten material to nozzle in the source.
Low Voltage Module	Supplies unregulated DC power to system electronics. (+20V at 10A and -16V at 0.9A.)
Relay Module	Controls the delivery of power to various modules and valves as triggered by the computer and the system switch.
Computer	Receives data from other modules and the Operator (through the terminal), and controls the operation of the μ PP.

2.3 DESCRIPTION OF μ PP PRINTED CIRCUIT BOARDS

The circuits used for controlling many of the Micro-Particle Processor system functions are mounted on custom printed circuit boards. For a listing of all P.C. drawing numbers, assembly numbers and board numbers, see Table 1-3 in Section 1. A description of the five (5) major P.C. boards is listed in Table 2-2 below:

TABLE 2-2
PRINTED CIRCUIT BOARD DESCRIPTIONS

<u>TITLE</u>	<u>DESCRIPTION</u>
Heater Controller	Regulates the temperatures of the source (reservoir and nozzle), as measured by thermocouples on the HV-ISO module, to the values set by the computer. Regulation is by controlling the heater duty cycle using the solid state heater power control relays on the HV-ISO module.

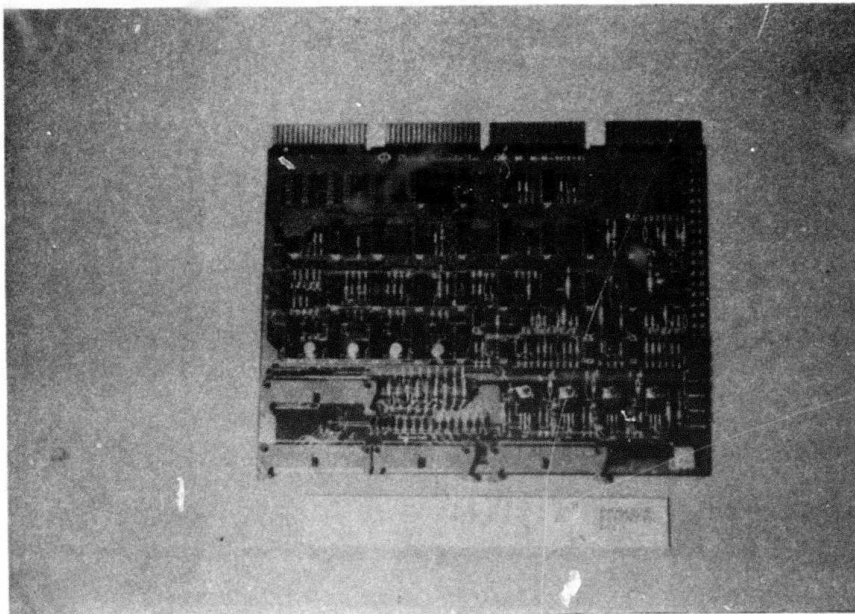
Feedpressure Controller	Controls the operation of the valves in the Feedpressure Manifold to regulate the feedpressure set by the computer and measured by the pressure transducer.
Tape Drive Stepper Motor Controller	Determines step speed of tape drive stepper motor; controls two speed levels: rapid and slow.
Tape Drive Frequency Counter	Frequency counter connected to the tape drive oscillator.
Relay Module	Contains relays for switching power to other system module components.

For reference purposes, the photographs in Figs. 2-4 and 2-5 identify the various printed circuit boards (relay module PC board not shown).

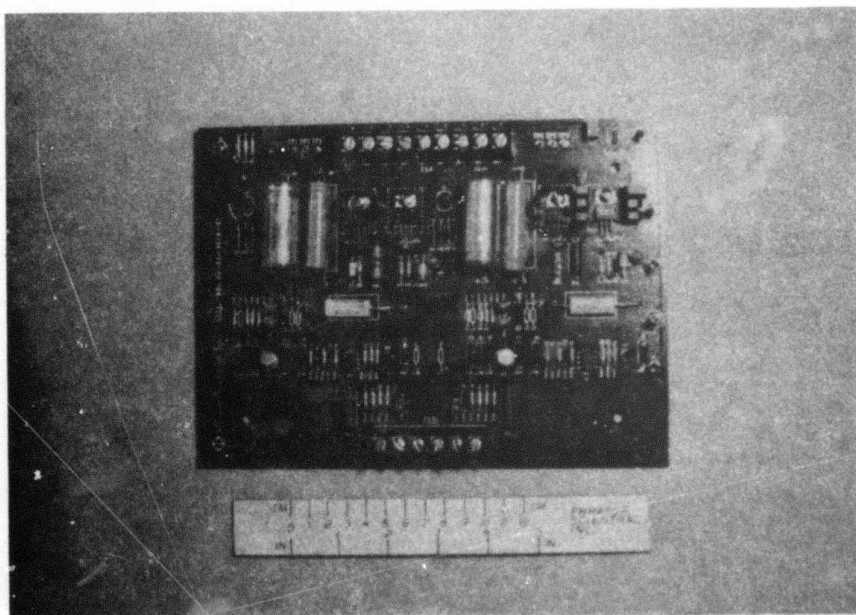
2.4 MICRO-PARTICLE SOURCE

The Micro-Particle Source is shown in Fig. 2-6, and a description of source components is listed in Table 2-3. Several different source configurations have been tested in the μ PP, and Fig. 2-6 is intended to familiarize the user with the basic source configuration and functional parts. The user will be required to handle the source for several operations. For reference, these operations are listed here along with the corresponding manual sections which describe the detailed handling procedures.

<u>OPERATION</u>	<u>MANUAL SECTION</u>
1. Unloading Reservoir Assembly from Micro-Particle Source	3.1
2. Installing Reservoir Assembly into Micro-Particle Source	3.2
3. Disassembly of Micro-Particle Source	5.2
4. Assembly of Micro-Particle Source	5.3

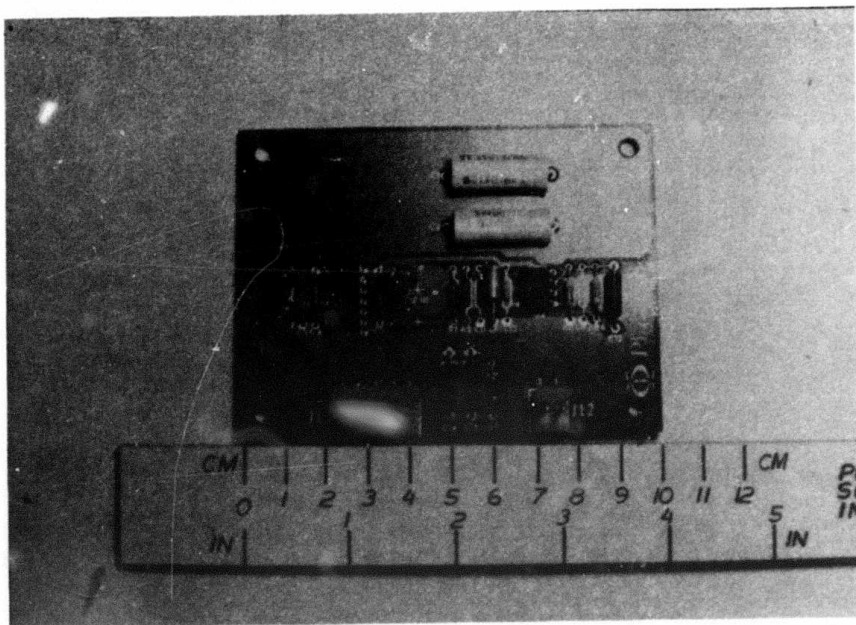


Quad Board

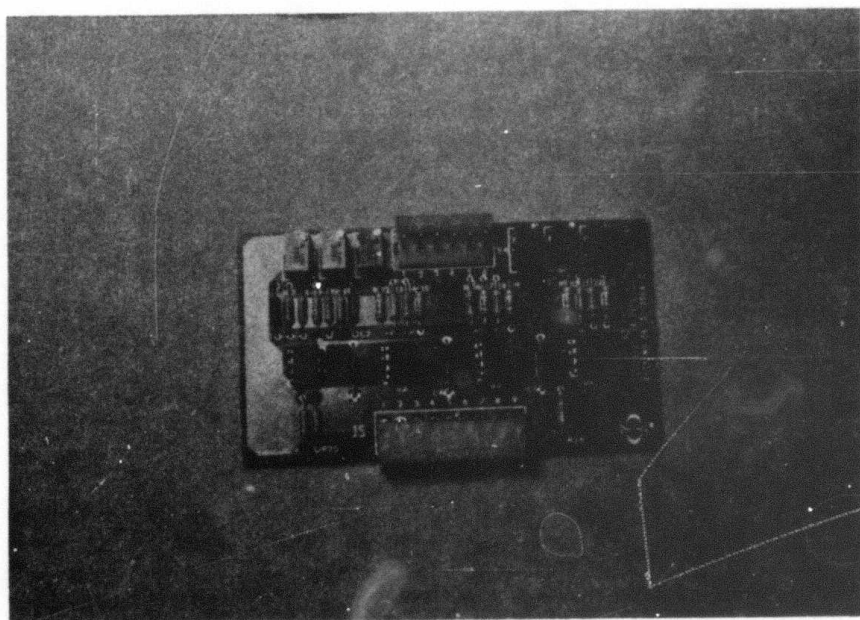


High Voltage Thermocouple

Fig. 2-4 Printed Circuit Boards



Source Current



Vacuum Set Point

Fig. 2-5 Printed Circuit Boards

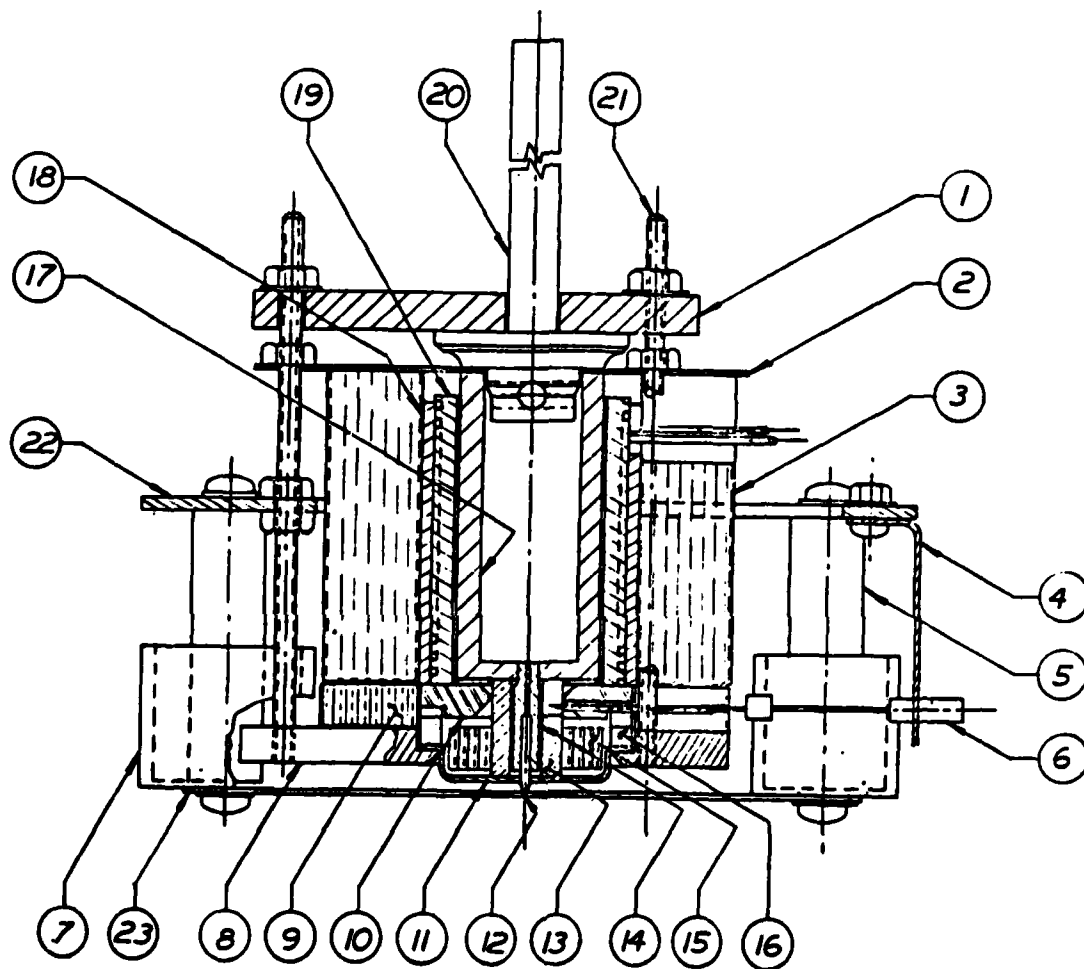


Fig. 2-6 Micro-Particle Source

TABLE 2-3
DESCRIPTION OF SOURCE PARTS

<u>NUMBER</u>	<u>PART NAME</u>
1	Reservoir Clamp Plate
2	Heat Shield Cover (Tantalum)
3	Reservoir Heat Shielding
4	Nozzle Heater Insulator Support
5	Extractor Insulator
6	Nozzle Heater Insulator
7	Extractor Insulator Shield
8	Bottom Clamp Plate
9	Nozzle Heat Shielding
10	Reservoir Bottom Support Ring
11	Nozzle Heat Shield Cover
12	Nozzle
13	Nozzle Heater Body
14	Nozzle Holder Feed Tube
15	Nozzle Heat Shielding
16	Ceramic Spacer
17	Reservoir Body
18	Reservoir Heater Insulator
19	Reservoir Heater Core
20	Feedpressure Inlet Cap
21	Support Post, CRES (3)
22	Support Ring
23	Extractor Electrode

Operations #1 and 2 above involve the routine loading and unloading of the ceramic reservoir assembly with the Micro-Particle source installed in the vacuum system. This feature allows for rapid turn-around times for processing and completing several powder runs. Allowing for normal heat up and cool down cycles, it is possible to conveniently and efficiently complete three (3) test runs in a single day.

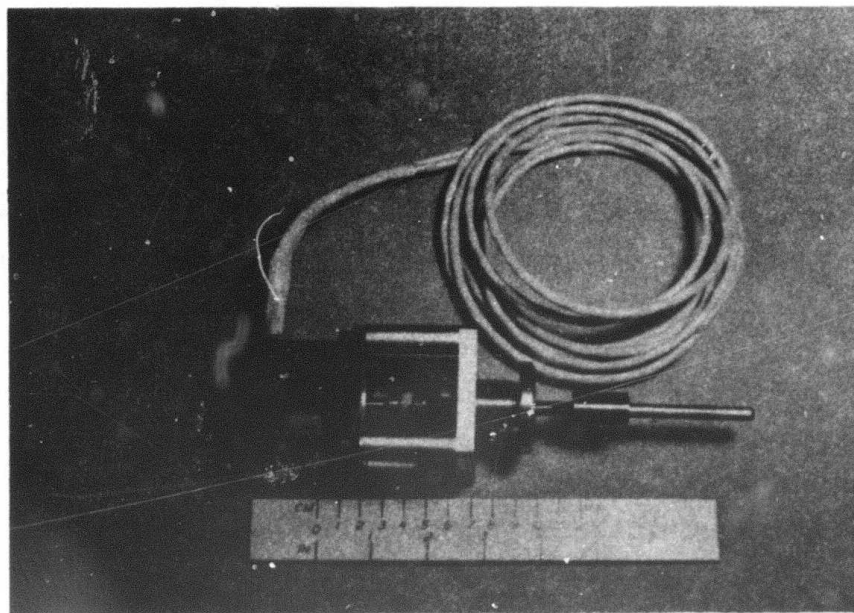
Operations #3 and 4 require the removal of the top plate with source assembly mounted and other μ PP structures for general source maintenance or replacement of components such as nozzle and/or reservoir heaters. Thermocouples can also be replaced during these operations, but is not required since the user can remove and install new thermocouples when the source assembly is still mounted in the vacuum chamber.

2.5 TAPE DRIVE ASSEMBLY

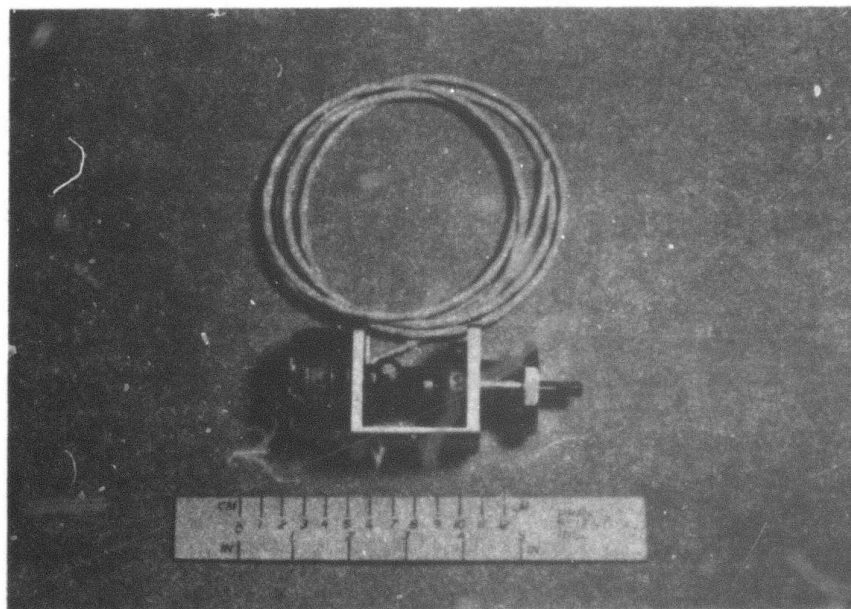
Various views of the tape drive assembly construction are shown in Fig. 3-3 and Fig. 3-4 of Section 3.3. For an identification of components used in the construction of the tape drive assembly, the user is referred to the parts list in Table 3-1. The tape drive system is operated by the tape drive stepper motor and shutter solenoid depicted in the photographs of Fig. 2-7. These components are mounted on the base flange at feedthrough locations BF1 and BF2 respectively (see Section 5.1, Fig. 5-2 for a visual layout of the base plate feedthrough locations). Details of the tape drive stepper motor and shutter solenoid construction are shown in Figs. 2-8 and 2-9.

2.6 VACUUM SYSTEM AND PUMP STACK

The pumping system used in the Micro-Particle Processor unit is shown schematically in Fig. 2-10. Manufacturer manuals with operational and maintenance instructions for major vacuum system components such as the diffusion pump, rough pump and cryo-baffle are supplied with this basic μ PP Users' Manual. Various component model and serial numbers are listed in Table 1-2.



Tape Drive Stepper Motor



Tape Drive Shutter Solenoid

Fig. 2-7 Tape Drive Base Plate Components

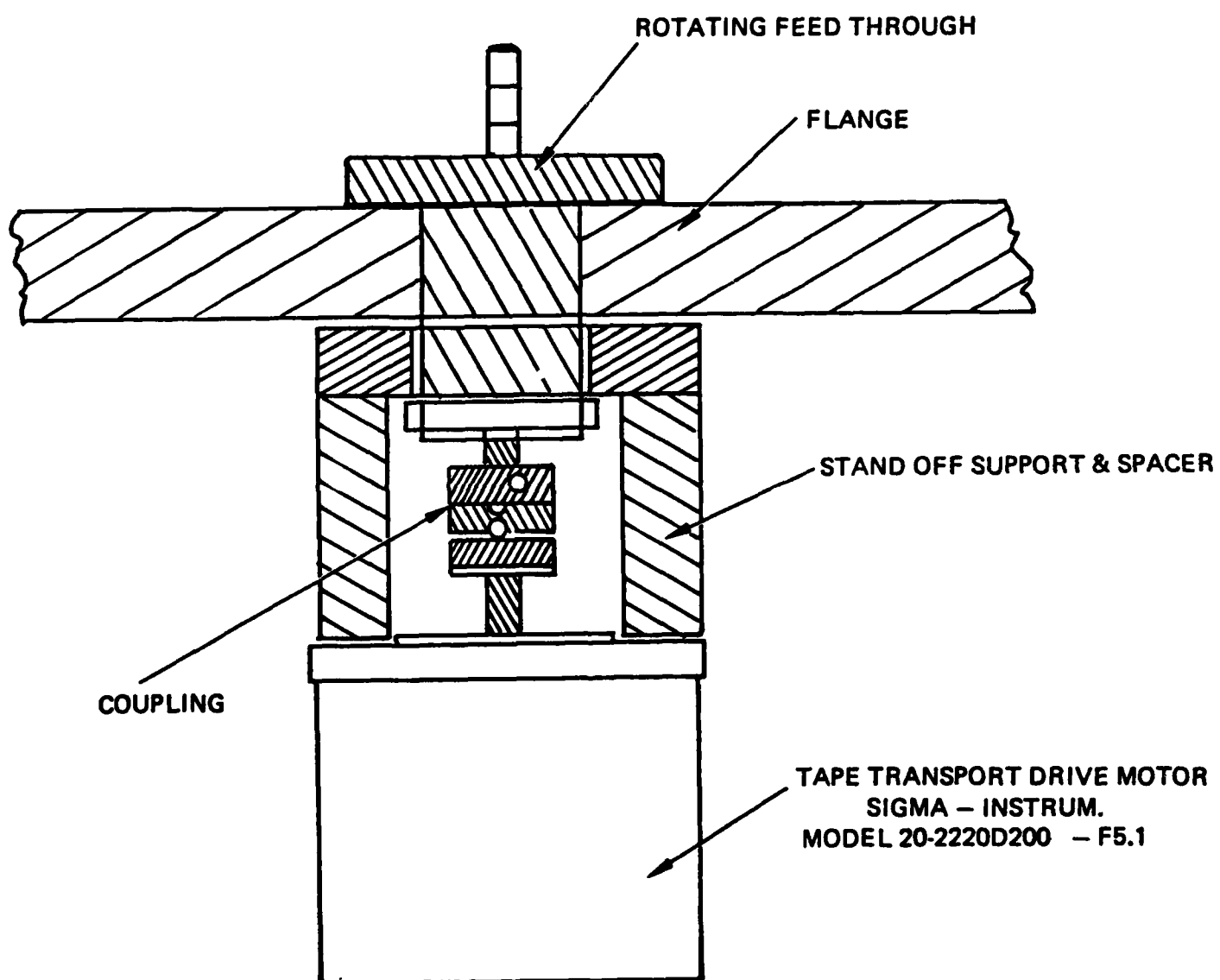


Fig. 2-8 Tape Drive Stepper Motor

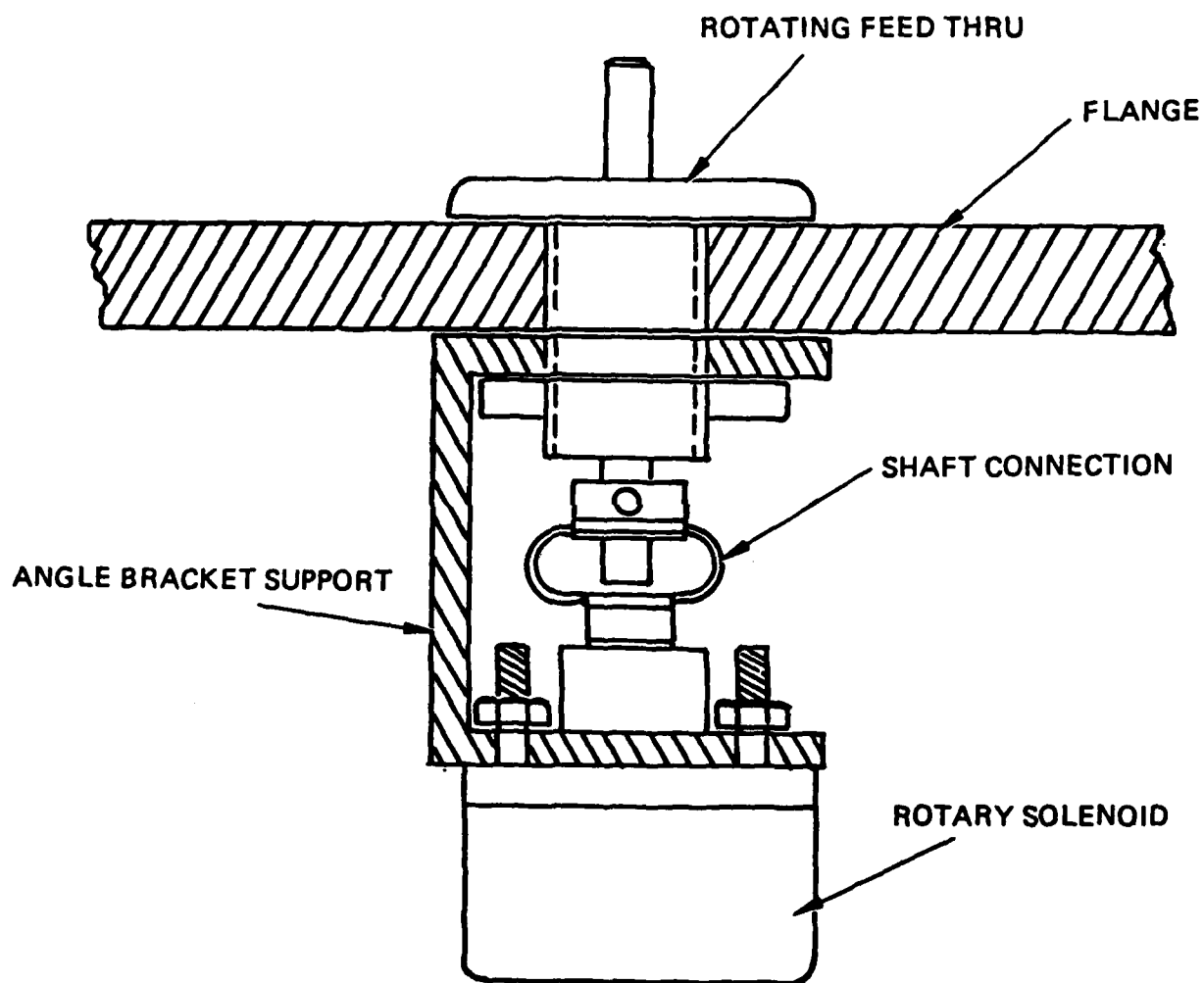


Fig. 2-9 Tape Drive Shutter Solenoid

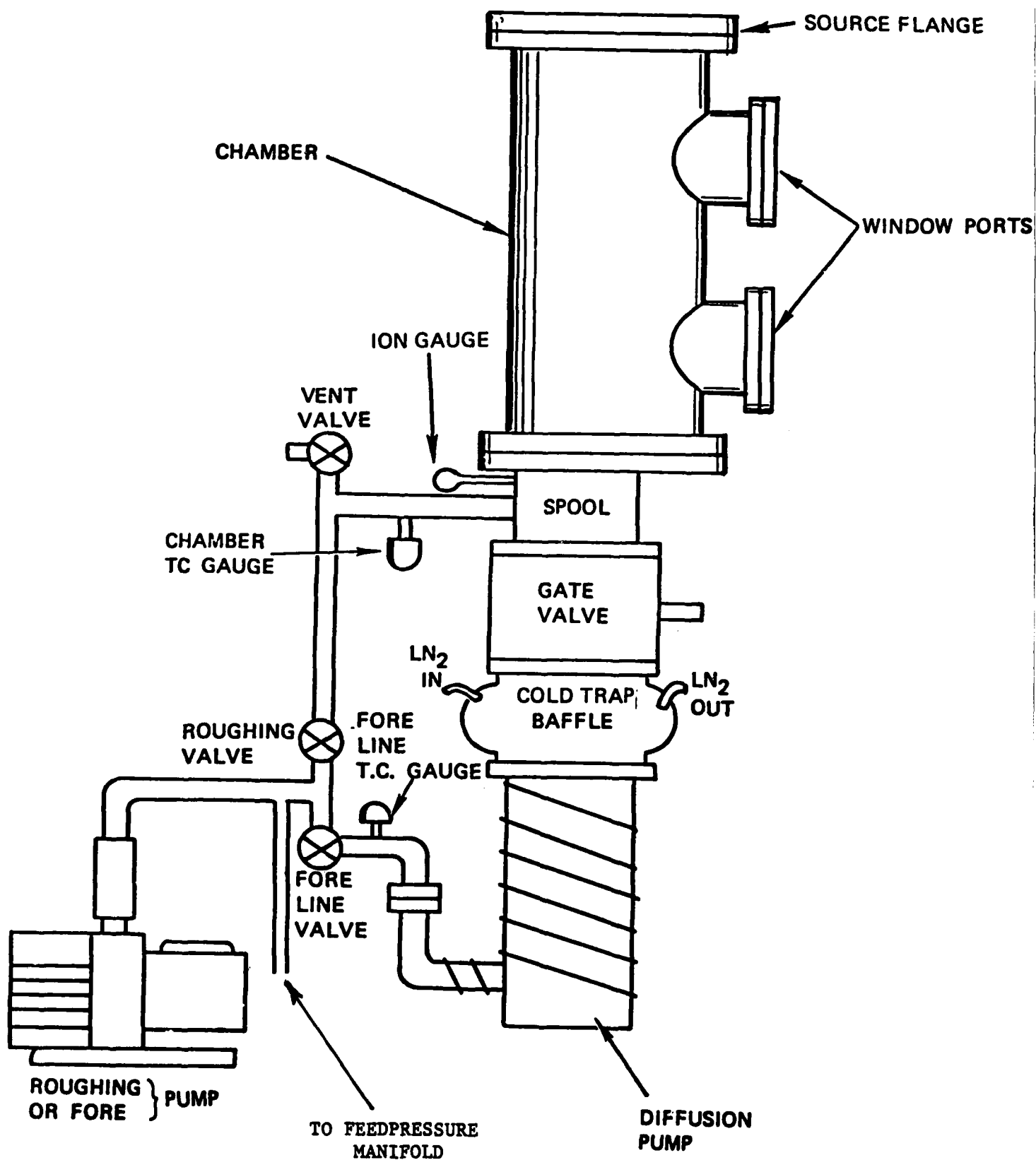


Fig. 2-10 Vacuum System and Pump Stack

2.7 FEEDPRESSURE MANIFOLD

An important sub-component of the μ PP vacuum system is the reservoir feed system module or feedpressure manifold shown in the photograph of Fig. 2-11. Connections for the rough pump and external gas supply are shown in the reservoir feed system valve schematic of Fig. 2-12. A separate manual detailing operational instructions for the Datametrics pressure transducer is also provided with this manual. (Refer to Table 1-2 for model and serial number identification.)

2.8 HIGH VOLTAGE ISOLATION MODULE

The physical layout of the high voltage isolation module discussed in Section 2.2 is shown in Fig. 2-13. A description of module components corresponding to the reference designations of Fig. 2-13 is listed in Table 2-4 below:

TABLE 2-4
HIGH VOLTAGE ISOLATION MODULE PHYSICAL LAYOUT (SEE FIG. 2-13)

Reference Designation	Description
1	Cooling Fan
2	EMI Shield Box Cover, 1/8" Thick Steel
3	1/8" Thick Al Static Shield Plate
4	High Voltage Thermocouple P.C. Board
5	1/4" Thick Al Heat Sink for Solid State Switches
6	3/4" Plexiglas Insulation Plate
7	High Voltage Isolation Transformer
8	Inductor
9	Limiting Resistor (2M Ω , 20 Watts)
10	Solid State Switch
11	Insulator
12	Metal Standoff

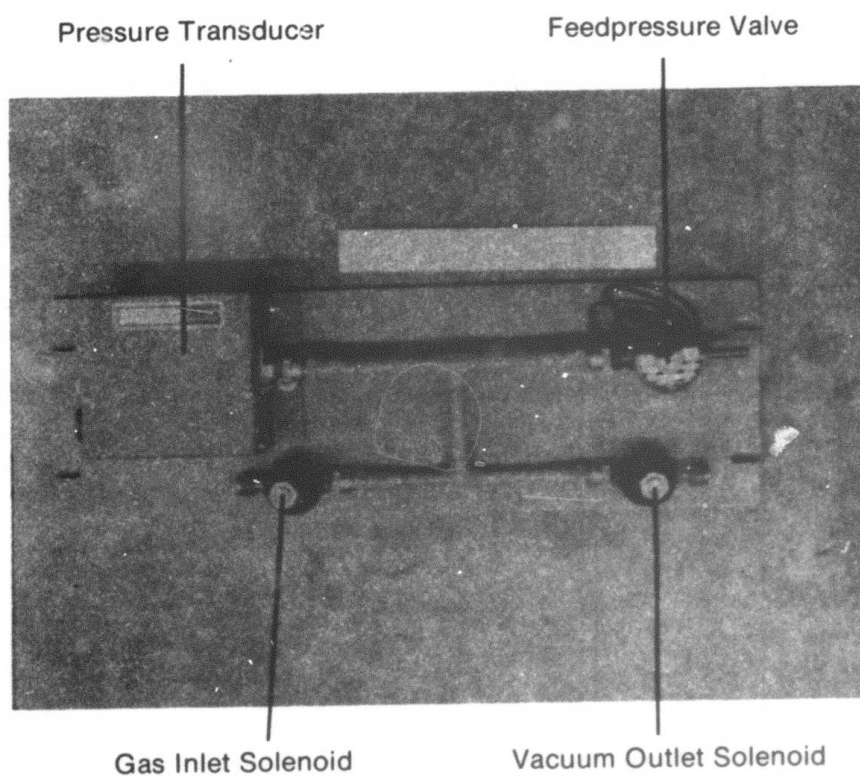


Fig. 2-11 Reservoir Feed System Module

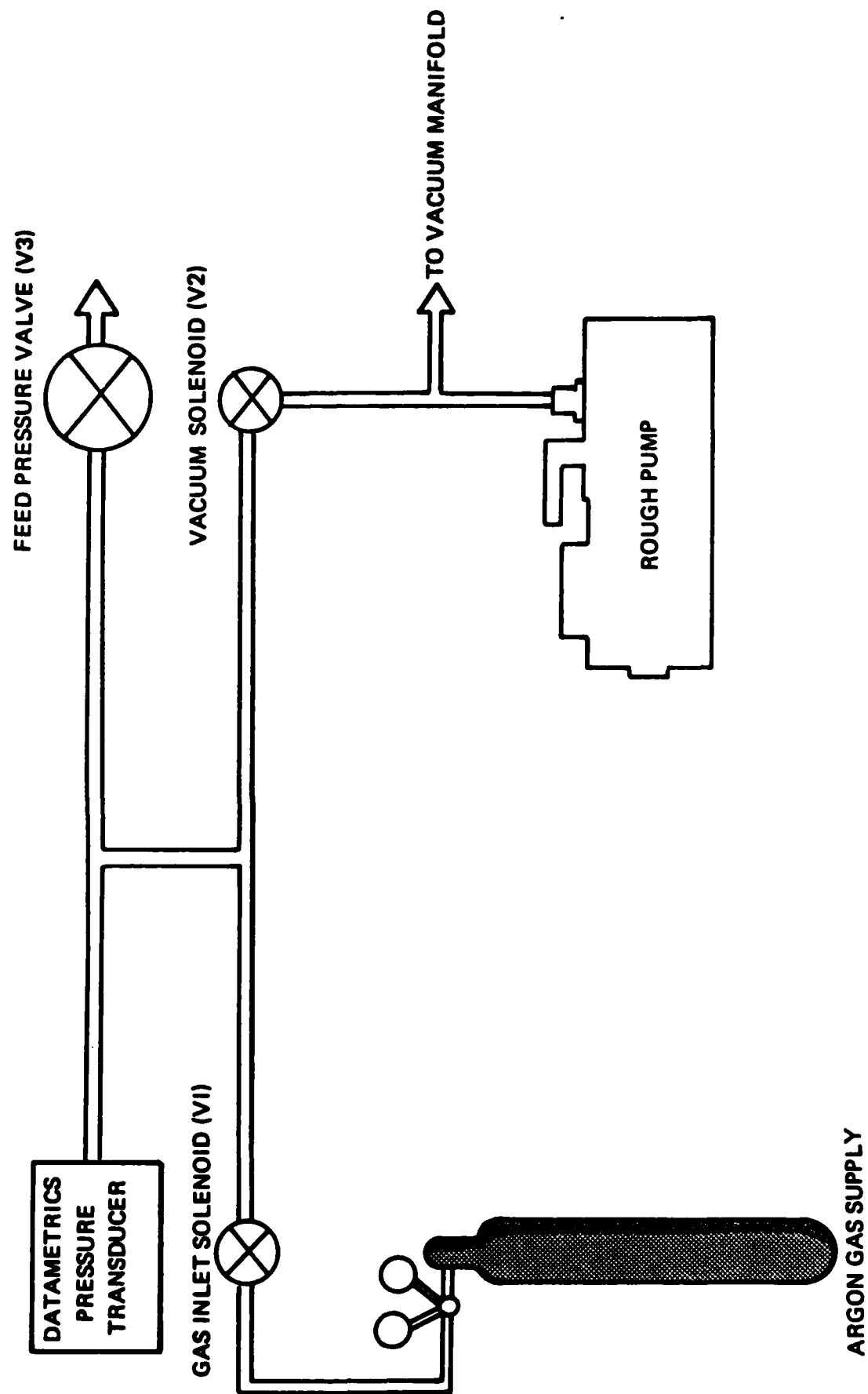


Fig. 2-12 Reservoir Feed System Valve Schematic

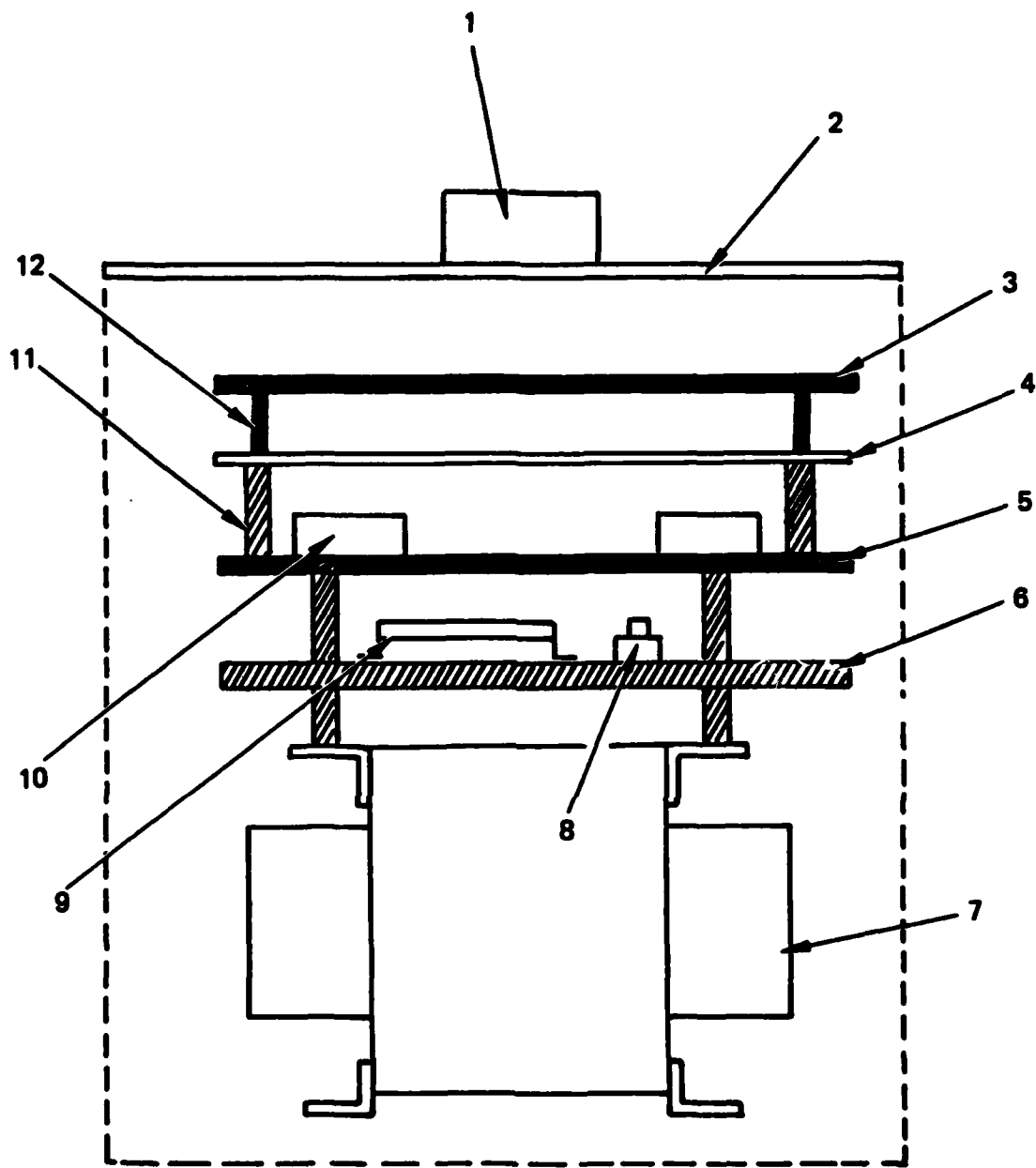


Fig. 2-13 High Voltage Isolation Physical Layout

External connections to the isolation module are shown in Fig. 2-14. The components within the high voltage module are enclosed in an EMI Shield Box made of 1/8" thick steel to suppress electromagnetic interference arising at this location during source arcing. This box must be in place at all times to prevent computer halting during arcing disturbances.

2.9 RELAY MODULE

The relay module discussed in Section 2.2 is shown in the photographs of Fig. 2-15 illustrating the connector locations on the left and rear panels. Figs. 2-16 and 2-17 detail the physical location of the panel connectors appropriately labeled with connector number assignments. Tables 2-5 and 2-6 summarize the relay module rear and left side connector functions respectively. A description of relay operations is listed in Table 2-7.

2.10 SUPPRESSION OF ELECTROMAGNETIC NOISE FROM μ PP

Elimination of both radiated and conducted EMI interference with the computer control system in the μ PP has required the installation of various preventive systems and suppression components. These elements are listed in Table 2-8 below:

TABLE 2-8
EMI SUPPRESSION COMPONENTS

-
- | | |
|----|---|
| 1. | High Voltage Isolation Module EMI Shield Box |
| 2. | Top Flange EMI Shield Box |
| 3. | L-C Filter Assembly |
| 4. | High Voltage Cover |
| 5. | Series Inductance in the Primary H.V. Line |
| 6. | Multiple Transient Suppressors on P.C. Boards |
-

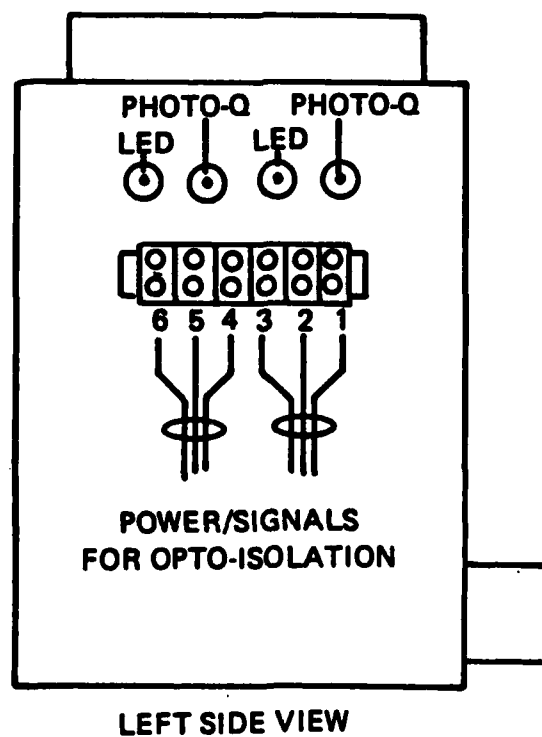
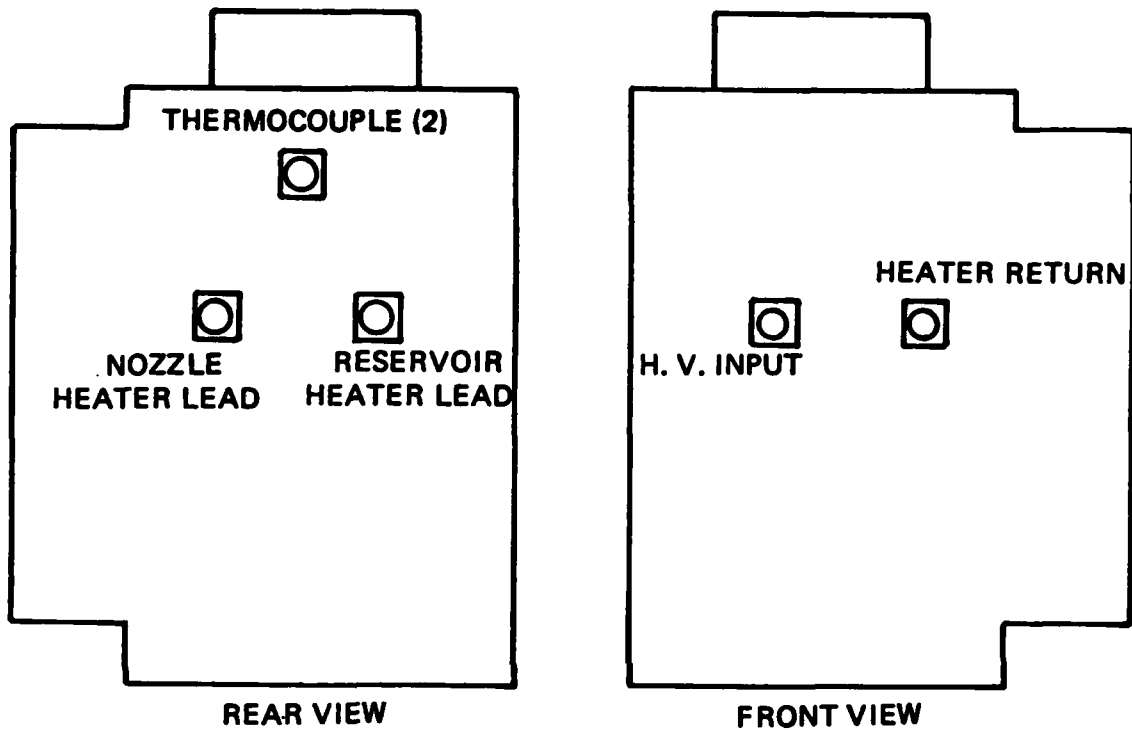
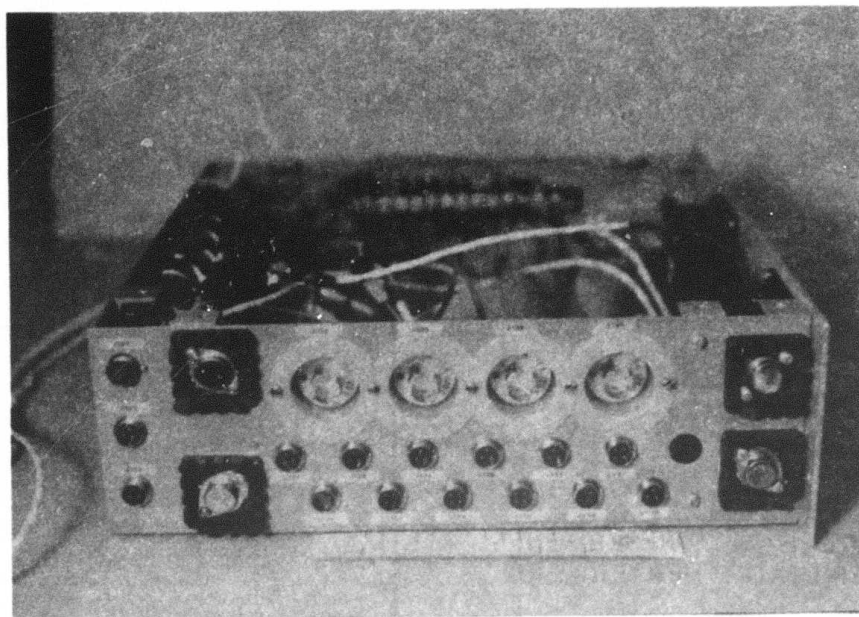
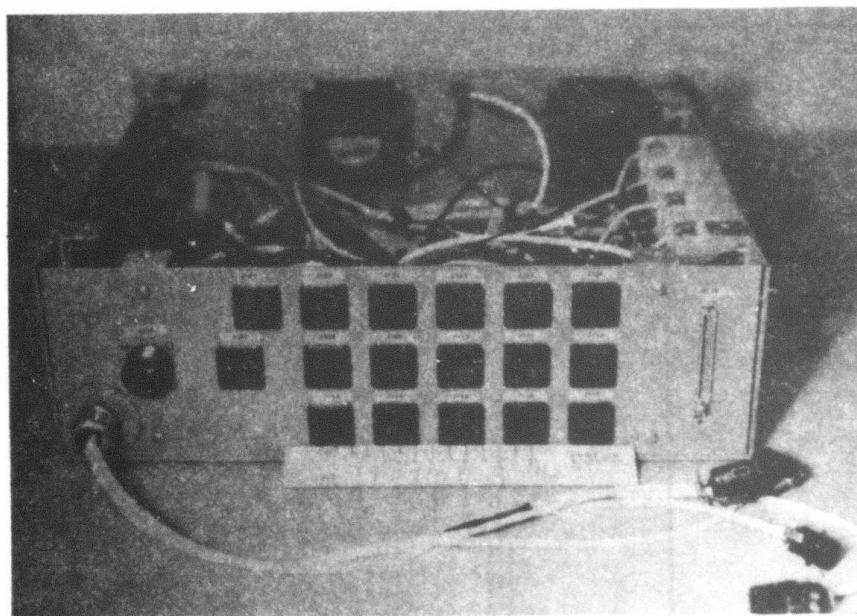


Fig. 2-14 High Voltage Isolation Module Connections



Relay Module Panel (Left Side)



Relay Module Panel (Rear)

Fig. 2-15 Relay Module Panels Showing Connectors

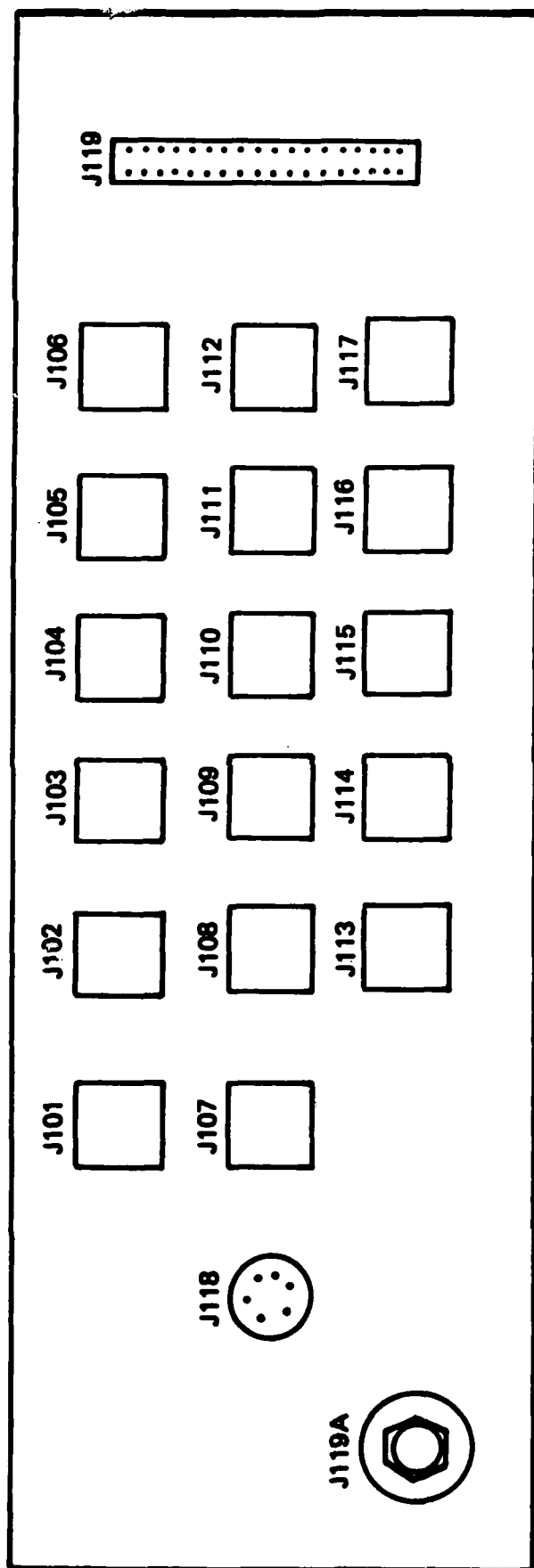


Fig. 2-16 Relay Module Panel (Rear)

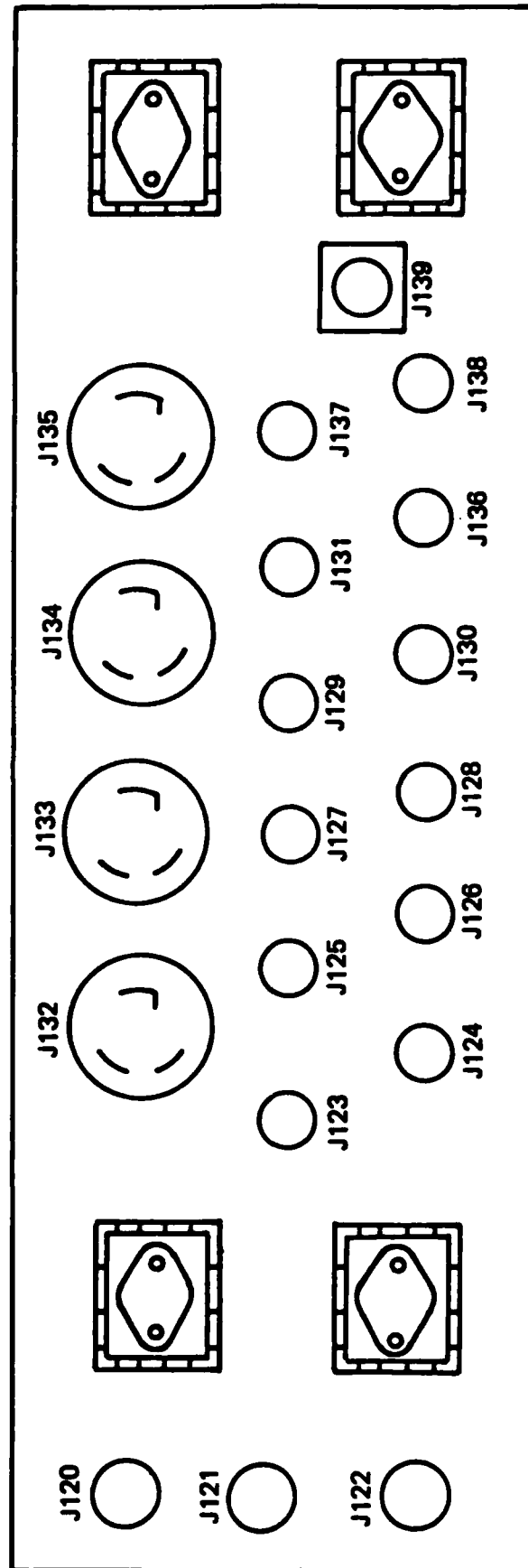


Fig. 2-17 Relay Module Panel (Left Side)

TABLE 2-5
RELAY MODULE PANEL (REAR)
Connector Designations

Connector Designations	Description
J101	Negative Logic Supply Power
J102	Positive Logic Supply Power
J103	Printer Power
J104	Computer Power
J105	Vacuum Ion Gauge Controller Power
J106	Video Monitor Power
J107	Keyboard Terminal Power
J108	Roughing Pump Power
J109	High Voltage Module Cooling Fan
J110	Main Cabinet Cooling Fan (Right Side Panel)
J111	Computer Cabinet Cooling Fan
J112	Main Cabinet Cooling Fan (Left Side Panel)
J113	Unassigned
J114	High Voltage Power Supply
J115	Diffusion Pump Heater
J116	Diffusion Pump Cooling Water Solenoid
J117	Spare Outlet (Not Switched)
J118	Reservoir/Nozzle Heater Transformer
J118-A	AC Return
J118-B	Output from K17 (H2) *
J118-C	Output from K9 (H3)
J118-D	Output from K10 (H4)
J118-E	Output from K16 (H5)
J118-F	Output from K11 (H6)

TABLE 2-5 Continued

J119	Computer to Relay Module Control Signal Cable
J119-A	Vacuum System Valve Control Outputs Output from K7 to Gate Valve Output from K12 to Roughing Valve Output from K20 to Vent Valve Output from K22 to Foreline Valve

* H2, H3, etc. are transformer tap number designations.

TABLE 2-6
RELAY MODULE PANEL (LEFT SIDE)
Connector Designations

Connector Designation	Description
J120	Output from J7 on Relay Module P.C. Board
J121	Logic Supply Output to Quad P.C. Board
J121-1	- 16V Unregulated DC
J121-2	Common
J121-3	+ 20V Unregulated
J122	Input from Logic Supplies
J123	Unassigned Logic Supply Output
J124	Sync Pulse Output for Nozzle/Reservoir Heater Controller
J124-1	White, to J11-1 Feedpressure Solenoid Input
J124-2	Black, to J11-2 Feedpressure Solenoid Input
J124-3	Red, to J11-3 60Hz Clipped Sine Wave Out
J124-4	Green, to J11-4 Tape Drive Sync Pulse Input
J125	Logic Supply Output to Pressure Transducer
J126	Feedpressure Solenoid Outputs (From J8)
J126-1	White, Gas Solenoid J8-1
J126-2	Black, Gas Solenoid to Output of VR1, +12VDC
J126-3	Red, Vacuum Solenoid to Output of VR1, +12VDC
J126-4	Green, Vacuum Solenoid J8-4
J127	Output to Source Current P.C. Board
J128	Output to Tape Drive Shutter Solenoid (From J9)

TABLE 2-6 Continued

J128-1	White, J9-1
J128-2	Black, J9-2
J128-3	Unassigned
J128-4	Unassigned
J129	Unassigned Logic Outputs
J130	Remote Ion Gauge On/Off Control Output
J130-1	White, Ion Gauge on (K15)
J130-2	Black, Ion Gauge on (K18)
J130-3	Red, Ion Gauge on (K18)
J130-4	Green, Ion Gauge on (K15)
J131	Input to Diffusion Pump Thermal Interlock (DPH) Relay (To J16)

Main Power

J132	Input to K1
J133	120 VAC to K6, K8-K11, K16, K17, J7-2
J134	120 VAC to K2, K19, J117
J135	120 VAC Output to Diffusion Pump Heater

J136-1	Unassigned
J136-2	Unassigned
J136-3	Unassigned
J136-4	Unassigned
J136-5	Ion Gauge Filament Status Input
J136-6	Unassigned
J137	Auxiliary Shutter (Not used)
J137-1	J9-3
J137-2	J9-4
J138	120 VAC Output to Feedpressure Valve

TABLE 2-6 Continued

J139	Tape Drive Stepper Motor Outputs
J139-A	To TS1-2,* ϕ A2 Driver Output
J139-B	To TS1-4, ϕ A2 Driver Output
J139-C	To TS1-3, ϕ B2 Driver Output
J139-D	To TS1-1 ϕ B2 Driver Output
J139-E	To 40 Ω Resistor Mounted on Inside of Relay Module Left Side Panel
J139-F	Unassigned

* TS = Terminal Strip

TABLE 2-7
RELAY DESCRIPTION

Relay Number	Description
K1	Output to J101-J107 and K5,7,12,14 20,22
K2	Output to all cooling fans except computer chassis and H.V. supply
K3	Unassigned
K4	Unused
K5	Output to J116
K6	Output to J114
K7	Output to J119A - Gate Valve
K8	Output to J113
K9	Output to J118-C
K10	Output to J118-D
K11	Output to J118-F
K12	Output to J119A - Roughing Valve
K13	Output to J115
K14	Output to Feedpressure Valve Solenoid
K15	Vacuum Ion Gauge Turn On Relay
K16	Output to J118-E
K17	Output to J118-B
K18	Vacuum Ion Gauge Turn Off Relay
K19	Output to J108
K20	Output to J119A - Vent Valve
K21	Unassigned
K22	Output to J119A - Foreline Valve
K23	Input from J16 (J131)

Item #1 consists of a 1/8" thick mild steel box and Item #2 is constructed from 1/16" thick mild steel plate. Item #2 was implemented to suppress electromagnetic interference arising from the source. This top flange EMI shield box shields all high voltage and thermocouple lines at the top flange feedthroughs. Item #1 suppresses the noise arising from the region of the high voltage isolation module. Item #3 consists of six (6) low pass filters constructed for the primary side of the high voltage isolation transformer. These low pass filters shown in Fig. 2-18 are contained within a separate compartment of the high voltage isolation module EMI shield box to minimize radiation coupling directly into the low pass filter assembly. The circuit diagram for the L-C filter assembly is shown in Fig. 2-19.

WARNING: Removal of any one of the suppressive components could lead to premature halting of the computer caused by high voltage arcing in the region of the source.

2.11 SOURCE TEMPERATURE MEASUREMENT AND CONTROL SYSTEM

In order to operate the source at the proper temperatures for a particular alloy material, a temperature measurement and control system is provided and mounted in the High Voltage Isolation module. Thermocouples are used to measure the temperatures of both nozzle and reservoir with their respective heater individually controlled. Since the source can be operated up to 20kV, the heater circuits must have appropriate electrical isolation. A functional block diagram of the source temperature measurement and control system is shown in Fig. 2-20.

The thermocouple amplifier accepts a low level signal from a thermocouple, provides amplification, signal conditioning, isolation and linearization. The high voltage isolation characteristics are obtained by frequency modulating the analog signal and transferring it across an air gap utilizing an LED and photo transistor. An analog output voltage is provided that linearly represents the measured temperature. The

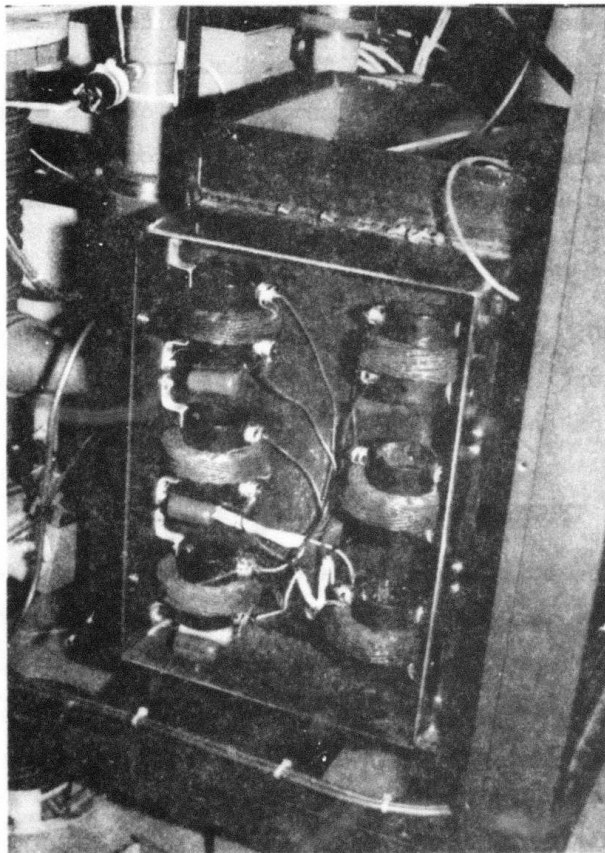
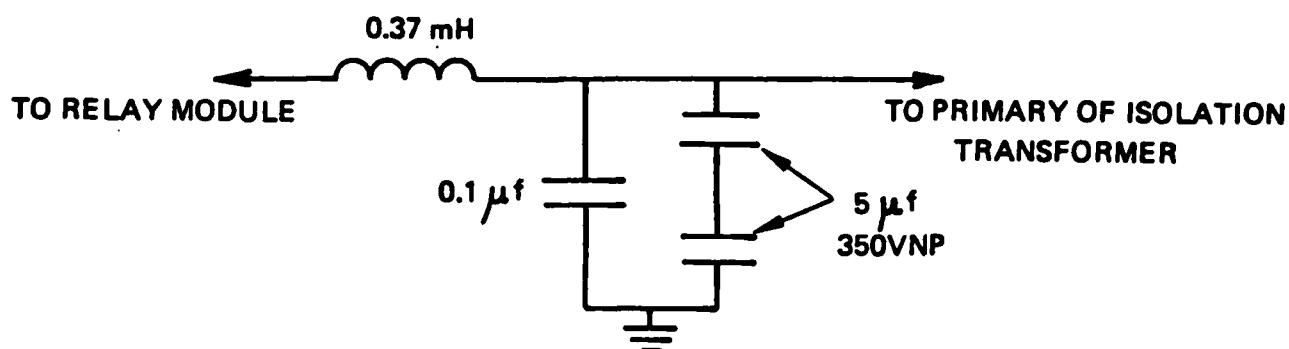
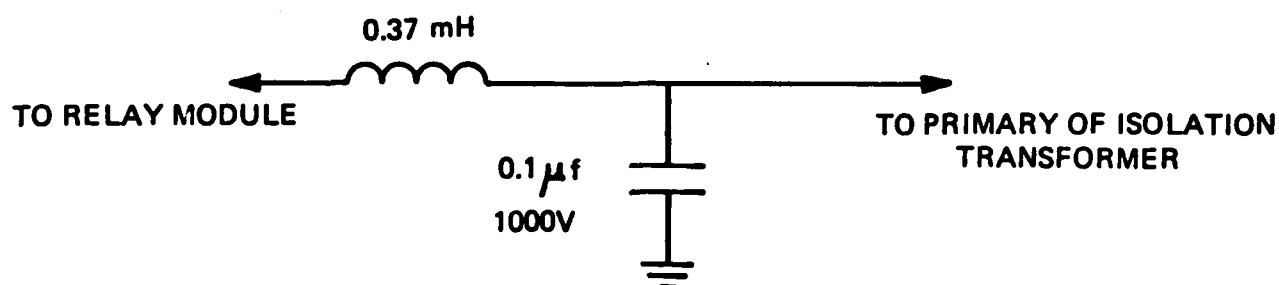


Fig. 2-18 L-C Filter Assembly



LEFT SIDE FILTER ASSEMBLY (3)



RIGHT SIDE FILTER ASSEMBLY (3)

RELAY NUMBER	TRANSFORMER TAP
LEFT SIDE	
H3	K9
H2	K17
H1	RETURN
RIGHT SIDE	
H6	K11
H5	K16
H4	K10

Fig. 2-19 High Voltage Isolation Module
L-C Filter Assembly

calibrated output produces one millivolt per degree C measured. This signal is used as input to the controller which compares it to the desired setpoint temperature and regulates the power to the heater by means of a solid state relay. The amplitude of the voltage that powers the heater is controlled by an autotransformer. A 20kV isolation transformer isolates the low voltage part of the system from the source. Referring to the block diagram of Fig. 2-20, a general description of the amplifier follows.

The input signal obtained from the thermocouple floating at high voltage is applied to a stable, precision amplifier with a low temperature coefficient of offset drift. This amplifier amplifies the thermocouple signal to a level of approximately one millivolt per degree input. A 0°C cold reference junction is included in the circuit that compensates for ambient temperature drift at the junction formed by the thermocouple wire connections to the amplifier input. The amplified analog signal is applied to the input of a voltage-to-frequency converter. The frequency output of the V-to-F converter drives a current switch that provides approximately 50mA current pulses to an LED. Power is supplied to this isolated input section of the amplifier by a power transformer whose breakdown voltage is in excess of 20kV. Regulated voltages are supplied by a dual plus and minus 12V regulator. The input section is totally isolated from power lines, earth ground, and the output section. It also has the capability to "float" to positive or negative potentials with respect to the output section or earth ground.

The frequency modulated pulses emitted by the infrared light emitting diode are sensed by a photodetector transistor. The amplified pulses are applied to a frequency-to-voltage converter. An output of one millivolt from the F-to-V converter represents approximately 1°C of the measured temperature. The current output pulses of the F-to-V converter are filtered by the two-pole filter and its associated components.

Linearization for Types C and K thermocouples is accomplished at an amplifier and at the switchable linearization components utilizing a five segment approximation for the desired nonlinear curves. Gain and offset adjustments are made at the amplifier at each linearization break point. The linear output of the amplifier is one millivolt per °C. The full scale capabilities of the system (maximum accuracy) are 1350°C for Type K, chromel/alumel, and 1650°C for Type C, tungsten-5% rhenium/tungsten-26% rhenium, thermocouples.

The temperature controller operates in a time proportioning mode which regulates the controller's duty cycle (ratio of time on to the total time) by the deviation between the measured and setpoint temperature.

2.12 MAIN CONSOLE CONNECTIONS

Various utilities interface with the μ PP via suitable connectors provided at the rear of the main console. These connections include compressed air, argon gas, electrical power and cooling water lines for the diffusion pump and vacuum chamber. (See Fig. 2-21 for the location of these connections.)

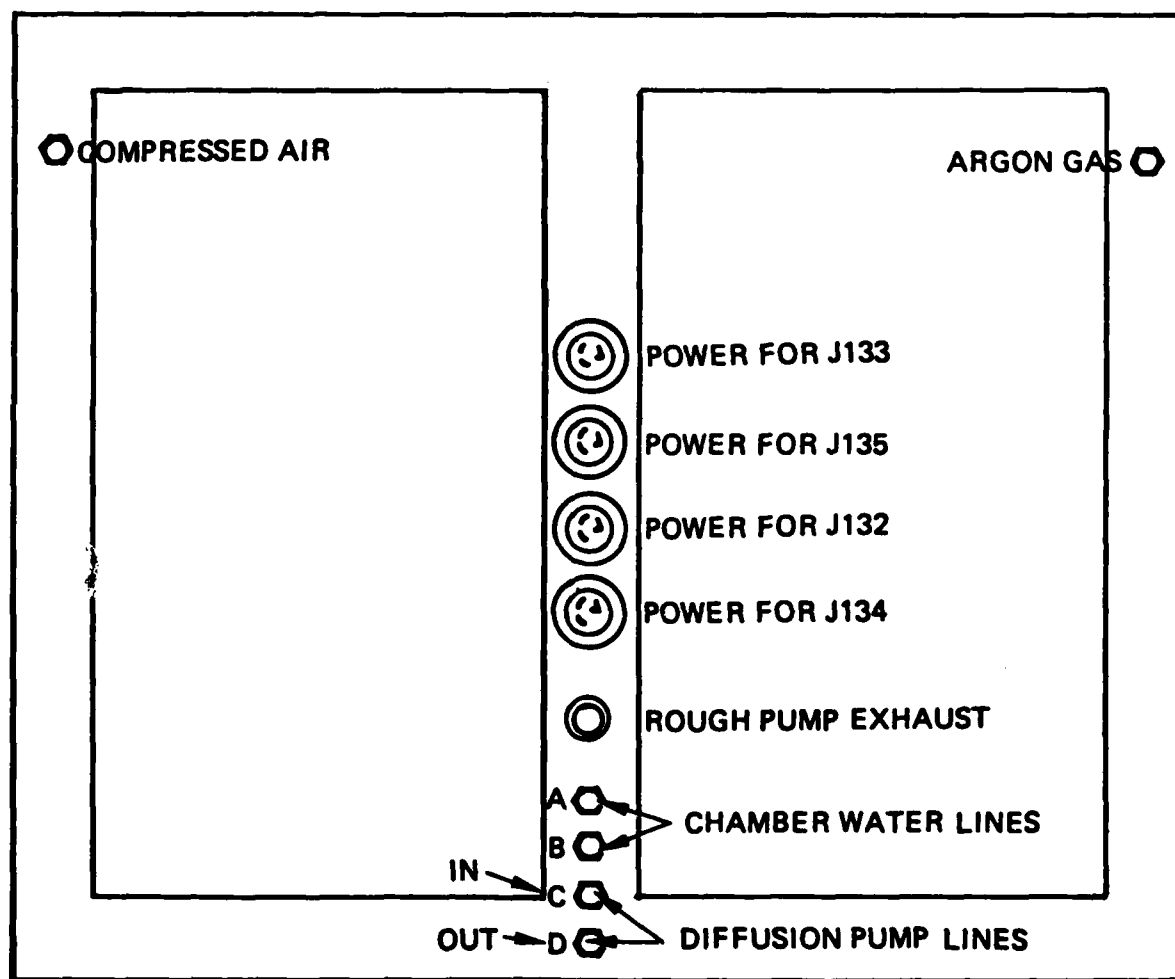


Fig. 2-21 Main Cabinet Connections (Rear)

SECTION 3.
PREPARATION FOR POWDER PROCESSING

3.1 UNLOADING RESERVOIR ASSEMBLY FROM MICRO-PARTICLE SOURCE

If the ceramic reservoir assembly has been previously removed, continue to Section 3.2 for instructions on how to install new reservoir assembly into the Micro-Particle Source.

1. Vent chamber as instructed by Shutdown Mode (see Section 4).
2. Open source vacuum port by loosening four (4) wing nuts on door latches, being careful not to dislodge O-ring.
3. Slide heat shield door open to expose source.
4. Loosen reservoir clamp thumb nut and rotate clamp plate counter-clockwise to disengage. Remove clamp plate completely.
5. Lift off feedpressure inlet cap from top of reservoir body. (Refer to Fig. 3-1 for identification of gas inlet and reservoir clamp components.)

NOTE

Do not flex bellows feed tube excessively.

6. Remove reservoir assembly by gently sliding straight up.

CAUTION

Verify that solidified metal has not bridged the gap separating nozzle tip and extractor plate before attempting to remove reservoir assembly. If a metal bridge has occurred, proceed to the next instruction.

7. Open tape drive port by loosening four (4) wing nuts on door latches being careful not to dislodge O-ring.
8. Using a small needle-nose plier and carefully reaching up through the tape drive port, remove the metal bridge.

GRAFOIL Gasket

Reservoir Clamp Plate

Inlet Cap

Thumbscrew

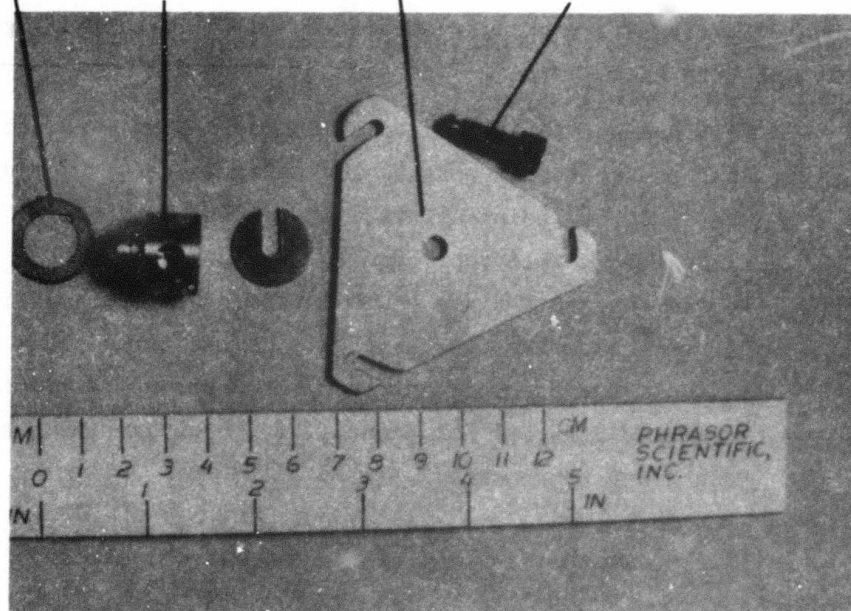


Fig. 3-1 Gas Inlet and Reservoir Clamp Components

9. Verify that the nozzle section of the Micro-Particle source is clear of solidified metal that could block removal from the source body.
10. Remove reservoir assembly by gently sliding straight up.

NOTE

On occasion, some metal deposit may remain attached to extractor plate after removing bridge. Proceed with Instruction #11.

11. Remove extractor plate from source by sliding free from retainer clips.
12. Replace with new extractor plate or previously removed extractor plate which has been carefully cleaned.

3.2 INSTALLING RESERVOIR ASSEMBLY INTO MICRO-PARTICLE SOURCE

1. Load reservoir assembly with metal charge.
2. Insert electrical contact strip (GRAFOIL) along the inside wall of the reservoir body to the bottom.
3. Bend upper (extending) portion of GRAFOIL strip perpendicular to the reservoir wall (see Fig. 3-2).
4. Carefully remove previous GRAFOIL gasket material, if any, from sealing surface of feedpressure inlet cap.
5. Reaching through source vacuum port, insert newly loaded reservoir assembly into Micro-Particle source body.

NOTE

Do not force reservoir assembly into source body. If, after careful manipulation, the reservoir assembly does not properly lower into position, the top flange may have to be removed to check for source misalignment.

6. Place feedpressure inlet cap onto top of reservoir assembly verifying that inlet cap seats level with the top sealing surface of the reservoir body.

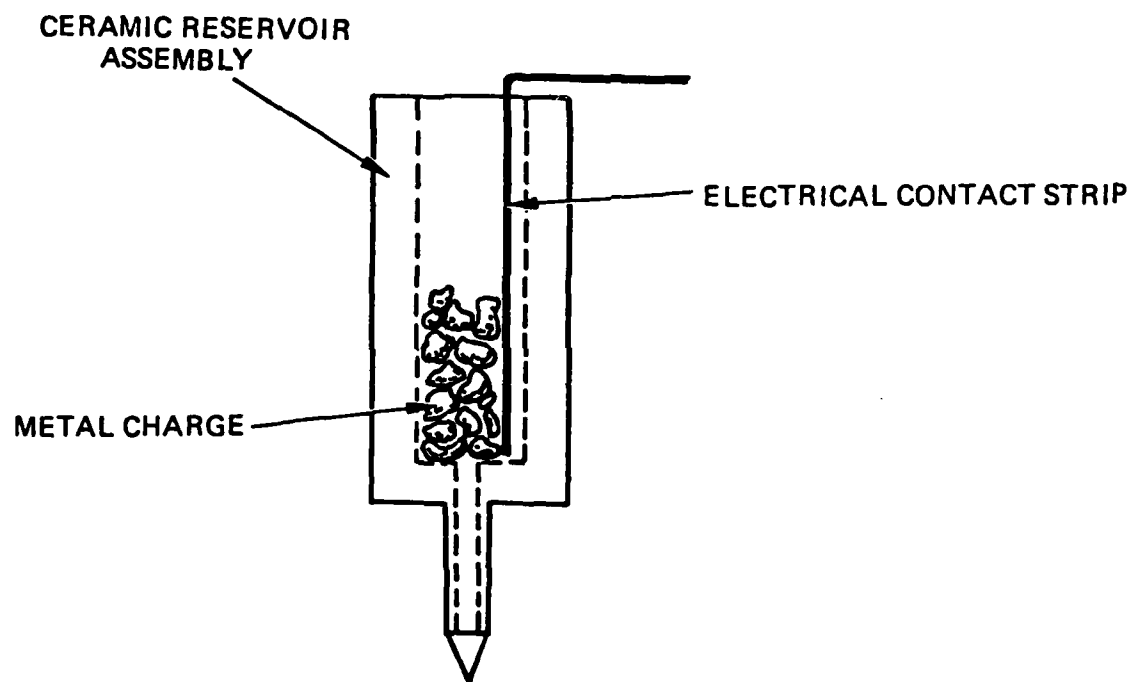


Fig. 3-2 Ceramic Reservoir Assembly

7. If the inlet cap does not seat level, carefully adjust the flexible feedline until cap and reservoir faces are parallel.
8. Lift inlet cap straight up a sufficient distance to insert GRAFOIL gasket in place.
9. Lower feedpressure inlet cap onto reservoir assembly.
10. Remount reservoir clamp plate in position and rotate fully clockwise to engage.
11. Tighten reservoir clamp plate thumb nut firmly using fingers only.
12. Slide heat shield door closed (option: shield door may be left open for improved source visibility for source operation that does not exceed 900°C).
13. After making sure that O-ring and source vacuum port surfaces are clean, close vacuum door.
14. Engage door clamps and tighten four (4) wing nuts.

After verifying that all external connections to the source have been properly made, the Micro-Particle source is now ready for operation.

3.3 LOADING THE TAPE DRIVE ASSEMBLY

If vacuum chamber has been previously vented for other operations. proceed to Instruction #2. If the tape drive assembly has not been previously unloaded, proceed to next Section 3.4.

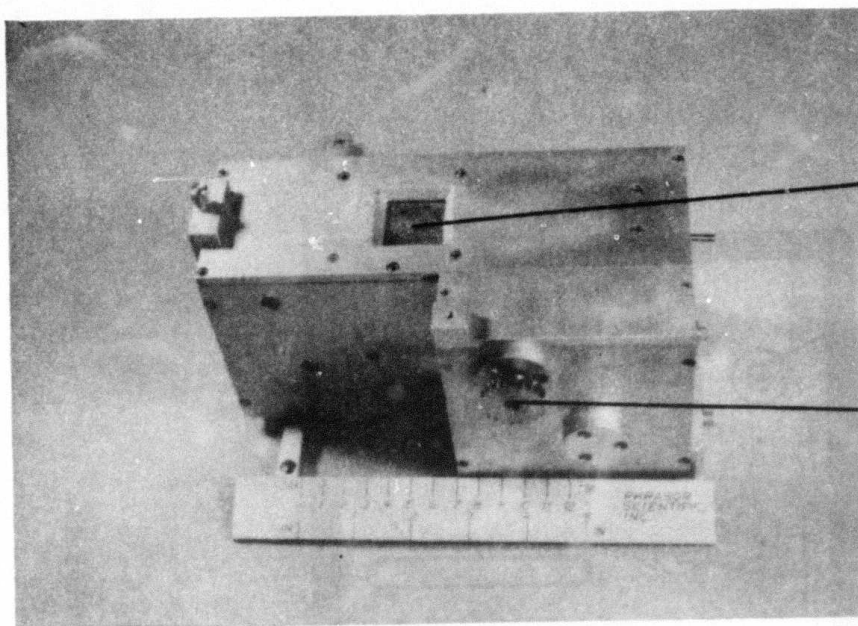
1. Vent chamber as instructed by Shutdown Mode.
2. Open tape drive port by loosening four (4) wing nuts on door latches, being careful not to dislodge O-ring.
3. Loosen and remove two (2) thumb screws from tape drive aperture plate and withdraw plate through port, being careful not to bend shutter.
4. Loosen thumb screw on the left side of the tape drive door.
5. Carefully disengage door from tape drive assembly while firmly holding both sides and remove through vacuum port.

6. While manually holding take-up spool rear plate in a locked position, unscrew take-up spool front plate (8) and remove. (See Fig. 3-3 and 3-4 for the location of tape drive assembly components. Refer to Table 3-1 for component description.)
7. Place roll of replicating tape on cover tape supply shaft (4).
8. Thread tape over "cover" tape guide roller (15) and insert into one of the slots on the take-up spool (6).
9. Place a second roll of tape on sample tape supply shaft (2).
10. Thread tape over "sample" tape guide roller (1) and through tape guide (16) located at the top of the tape drive assembly box (see Fig. 3-4).
11. While pressing the release bar (10) down, continue threading the tape between the capstan (14) and pinch roller (13).
12. Thread tape over the take-up spool (6) and insert into remaining slot of the take-up spool (6).
13. To remove slack in sample tape, press down on release bar (10) and carefully wind sample tape supply shaft (2) clockwise.
14. Remount and fasten front plate (8) by screwing onto take-up spool (6), being careful to hold take-up spool rear plate in a locked position.

CAUTION

To prevent tape drive assembly components from disengaging, do not allow the take-up spool (6) to rotate clockwise when mounting or removing take-up spool front plate (8).

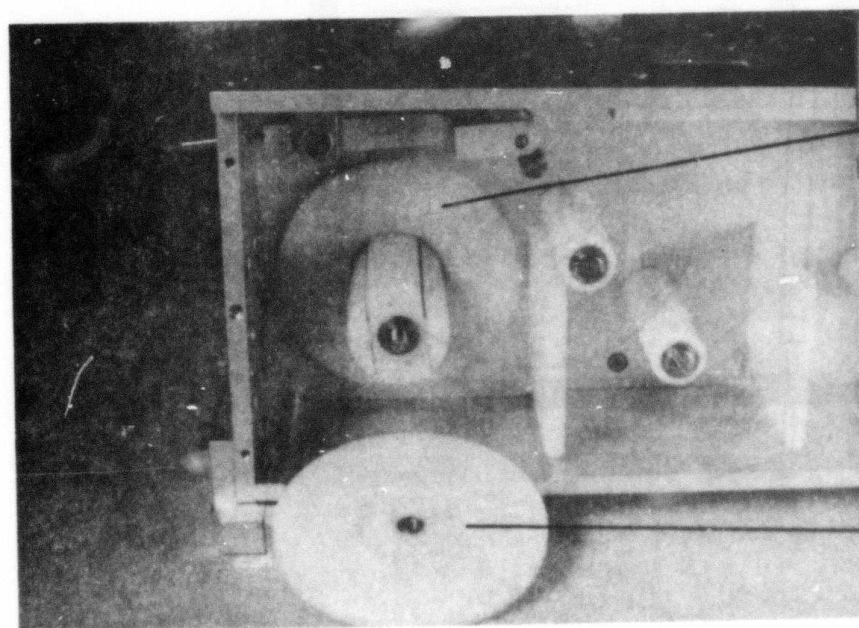
15. Being careful to align the right side guide pins, replace the tape drive door. Gently tighten the thumb screw on the left side of the door to hold.
16. Replace tape drive aperture plate (11) and gently fasten using two (2) thumb screws provided.



Sample Aperture

Capstan Sprocket

Top and Rear View



Take-up Spool Rear Plate

Take-up Spool Front Plate

Left Front View

Fig. 3-3 Tape Drive Assembly

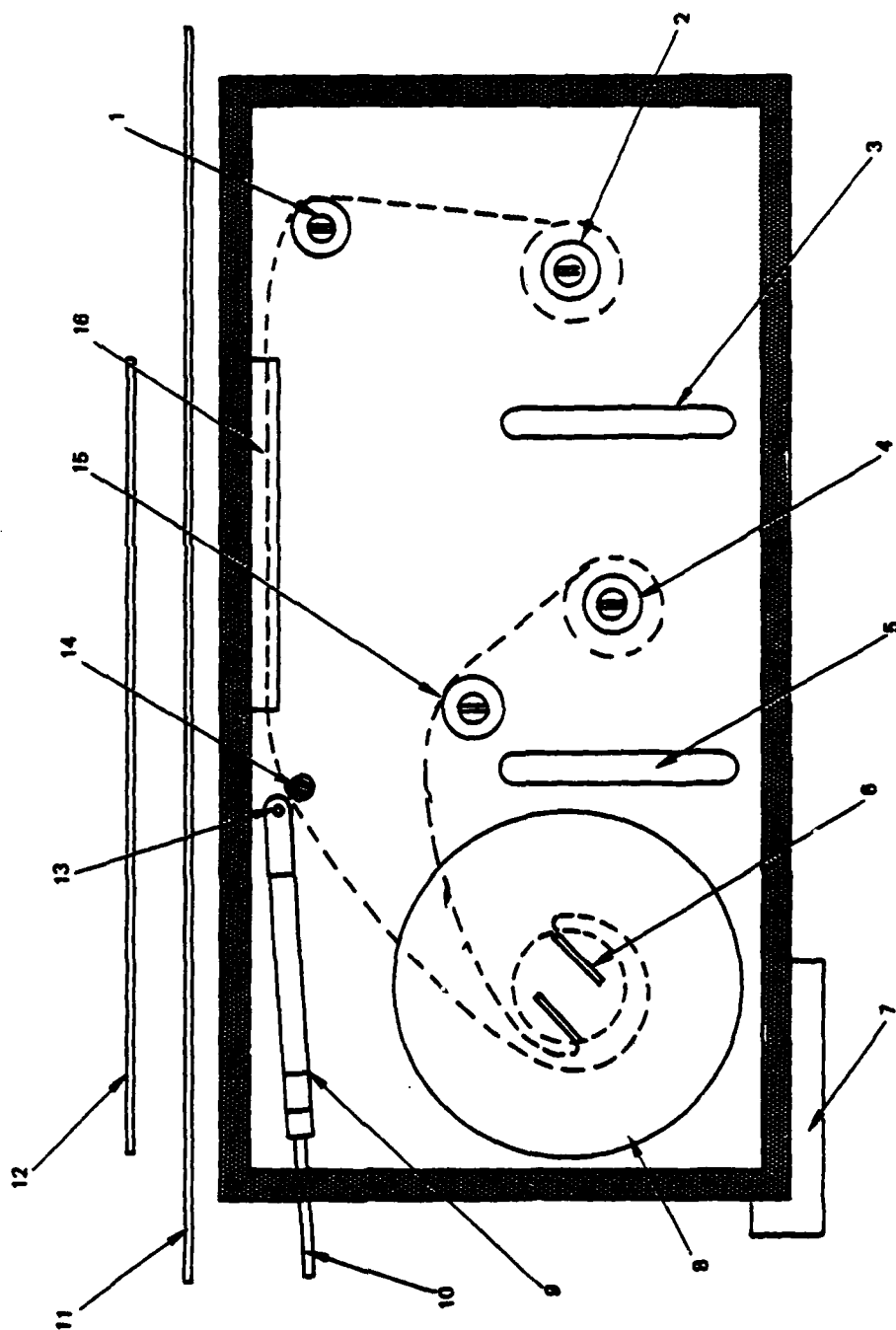


Fig. 3-4 Tape Drive Assembly Schematic

TABLE 3-1
TAPE DRIVE ASSEMBLY

Reference Designation	Description
1	Sample Tape Guide Roller
2	Sample Tape Supply Shaft
3	Tape Separator
4	Cover Tape Supply Shaft
5	Tape Separator
6	Take-up Spool
7	Tape Drive Assembly Mounting Bracket
8	Front Plate, Take-up Spool
9	Pinch Roller Assembly
10	Release Bar
11	Tape Drive Aperture Plate
12	Tape Drive Shutter
13	Pinch Roller
14	Capstan
15	Cover Tape Guide Roller
16	Tape Guide

3.4 UNLOADING THE TAPE DRIVE ASSEMBLY

Follow Instructions #1 through 5 in the previous section for venting chamber and removal of tape drive door.

1. While holding the take-up spool rear plate in a locked position, unscrew take-up spool front plate (8) and remove.
2. Advance sample tape to the left several inches (6") by clasping capstan tooth sprocket with fingers and rotating shaft.

NOTE

This advances tape with collected samples between take-up spool (6) and sample aperture onto take-up spool.

3. Being careful to hold wound tape on take-up spool (6), cut both sample and cover tapes near take-up spool.
4. Remove sample and cover tape roll by firmly holding roll and slide off the end of the take-up spool (6).

SECTION 4.
OPERATION OF THE MICRO-PARTICLE PROCESSOR

4.1 GENERAL DIRECTIONS

1. Capitals or lower case letters are interchangeable in all cases.
2. Single quotes are used to indicate exact wording appearing on the video monitor screen, e.g. 'Word or Phrase'.
3. Double quotes are used to indicate exact operator input. e.g., "Y" or "N".
4. "Return" refers to the "Return" key rather than typing the letters "return".
5. Footnotes are indicated in parenthesis as follows: (SM1), (SVP2), etc.
6. Temperatures are always in degrees Celcius.
7. If a mode is exited before completion, re-entry will usually be at the point of departure. The exceptions occur when tests or reconfirmation are necessary to insure proper operation.
8. All references to right, left or rear of the Micro-Particle Processor will be oriented with the operator standing in the operating position facing the video screen.
9. Questions and statements in this section will occur in the order presented by the computer. Statements and questions which do not apply will be skipped, e.g., if the chamber pumps down satisfactorily, the 'Possible Leak' message will be skipped.

4.2 TURNING ON μ PP

1. Turn on main power switch located to the right of the video screen.
2. Insert floppy disc with its label facing left. Close disc

door drive.

3. Briefly press blue disc boot switch located on computer panel lower right hand corner.
4. Wait for STANDBY MODE to appear on the video screen.

4.3 STANDBY MODE (See Fig. 4-1)

1. 'Date' and 'time'

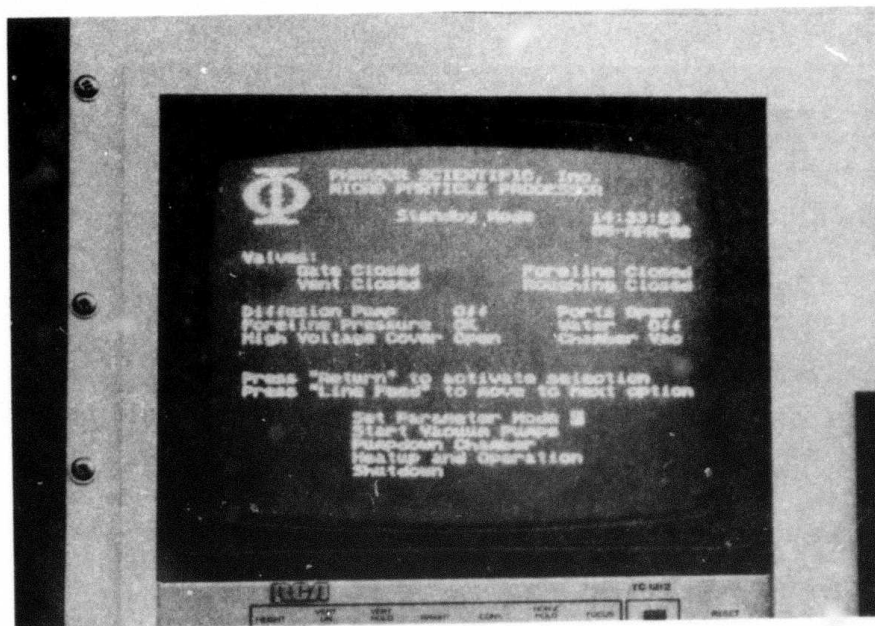
- A. At initial turn-on, the date will not be displayed and the time will indicate the length of time from the last disc boot operation.
- B. Operator changes are made in PARAMETER SETUP MODE.
- C. The computer will maintain the correct date and time until the computer is turned off or until the disc boot switch is pressed.

2. 'Valves'

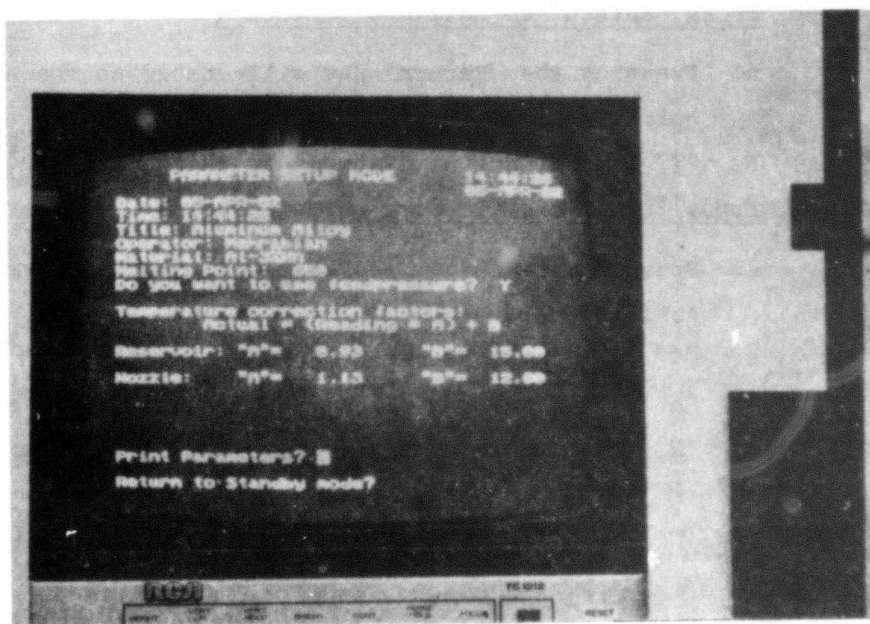
- A. The current status of each valve will be displayed.
 - a. 'open'
 - b. 'closed'
- B. Updating will occur automatically.

3. Status of Other Components

- A. 'Diffusion pump'
 - a. 'off'
 - b. 'on'
 - c. 'HOT' (will be in reverse video). The pump has overheated, the safety switch is tripped. The switch must be reset. See footnote (SML) at the end of this section for resetting procedure.
- B. 'Foreline pressure'
 - a. 'high'
 - b. 'ok'
- C. 'High Voltage Cover'
 - a. 'closed'



Standby Mode



Parameter Setup Mode

Fig. 4-1 Video Screen Displays

- b. 'open'
- D. 'Ports'
 - a. 'closed'
 - b. 'open'
- E. 'Water'
 - a. 'off' water solenoid valve closed.
 - b. 'on' water solenoid valve open
 - c. This will indicate when water should be off or on if water is controlled manually.
- F. 'Chamber Pressure'
 - a. 'Vac'
 - b. 'Atm'

4. List of Selectable Modes

Set Parameter

Start Vacuum Pumps

Pumpdown Chamber

Heatup and Operation

Shutdown

5. Press "Return" to Activate Selection

- A. Pressing the "Return" Key will change to the selected option screen.
- B. "Y" is the same as "Return" in this case.

6. Press "Line Feed" to Move to Next Option

- A. Pressing the "Line Feed" Key will move the cursor down one position.
- B. "^" will move the cursor up one position.
- C. "N" is the same as "Line Feed" in this case, except an "N" will remain on the screen, having no other effect.

4.4 PARAMETER SETUP MODE (See Fig. 4-1)

NOTE

All entries in this mode must be terminated by pressing the "Return" Key.

1. 'Date'

- A. If the date is not being displayed in the upper right hand portion of the screen, it must be entered before you can continue.
- B. The date is entered in standard computer format. e.g., "23-Feb-82".
- C. Changes or corrections can be made at any time by returning to this point.

2. 'Time'

- A. The time must be entered at least once after the computer has been turned on to continue past this point.
- B. The time is entered in standard computer format, example: "23:45[36]", using 24 hour notation with seconds being optional.
- C. Changes or corrections can be made at any time by returning to this point.

3. 'Title', 'Operator', and 'Material'

- A. Any combination of numbers, letters or symbols can be used.
- B. Maximum length is to the end of the line.
- C. These three lines may be used for special notes or procedures if desired as they have no effect on the actual operation and are for reference only.

4. 'Melting Point'

- A. Numbers only from 0 to 1300, degrees C are implied.
- B. Entry has no effect on operation and is for reference only.

5. 'Do you want to use feedpressure?'

- A. If "Y", then "Return" is pressed. The feedpressure valve will be open during HEATUP and OPERATION. All options

requiring feedpressure will be available.

- B. If "N", then "Return" is pressed and the feedpressure valve will be closed during HEATUP and OPERATION. All options requiring feedpressure will be unavailable. This option should only be used with the alternate reservoir clamp. See footnote SPML.

6. 'Temperature Correction Factors'

These are values used to compensate for the error because the thermocouple is not located at the desired point of measurement.

- A. $\text{Actual} = (\text{reading} * A) + B$, for both Nozzle and Reservoir where:
 - a. 'Actual' is the temperature at the thermocouple junction.
 - b. 'Reading' is the value displayed on the screen during HEATUP and OPERATE MODE.
 - c. 'A' is the scaling factor, which may have a value from 0.5 to 2.0.
 - d. 'B' is the offset value, which may have a value from -200 to 200.
- B. Large values of 'B' should not be necessary and 'B' is only used to compensate for slight offsets in the electronics between calibration intervals.

WARNING

Severe damage may result if the actual nozzle or reservoir temperature exceeds 1350°C. This condition can occur when high temperatures are used with values of 'A' greater than 1.00 and/or when 'B' is a large positive value. For example, with 'A' = 1.25 and 'B' = 200 ('B' is generally < 50), setting a temperature of 1150°C will give an actual temperature at the thermocouple of 1637°C.

7. 'Print Parameters'

- A. Pressing "Y" or "Return" will produce a written record of all currently displayed parameters (everything on the

video screen above this line).

- B. Pressing "N" will skip this option.
- C. Any number of copies may be made. Each will appear at the head of its own 8 $\frac{1}{2}$ x11 sheet. SET PARAMETER MODE may be re-entered at a later time for additional copies.

8. 'Return to Standby Mode?'

- A. Pressing "Y" or "Return" will return to STANDBY MODE and replace the previous parameter values with the current values in the permanent parameter file on the floppy disc.
- B. Pressing "N" will position the cursor at the top of the screen in front of 'Date'.
- C. Pressing "Escape" at this time or at any other point in SET PARAMETER MODE will also return to STANDBY MODE but will not replace the previous parameter values with the current values on the floppy disc. They will be maintained in temporary memory for immediate use only. They can be permanently stored by returning to this question and pressing "Y" or "Return".

4.5 STARTUP VACUUM PUMPS (See Fig. 4-2)

Only valid questions will be asked. (e.g., if the roughing pump is already on, the question, 'Turn on Roughing Pump?', will not be asked.)

1. 'Press "Escape" to return to Standby Mode'

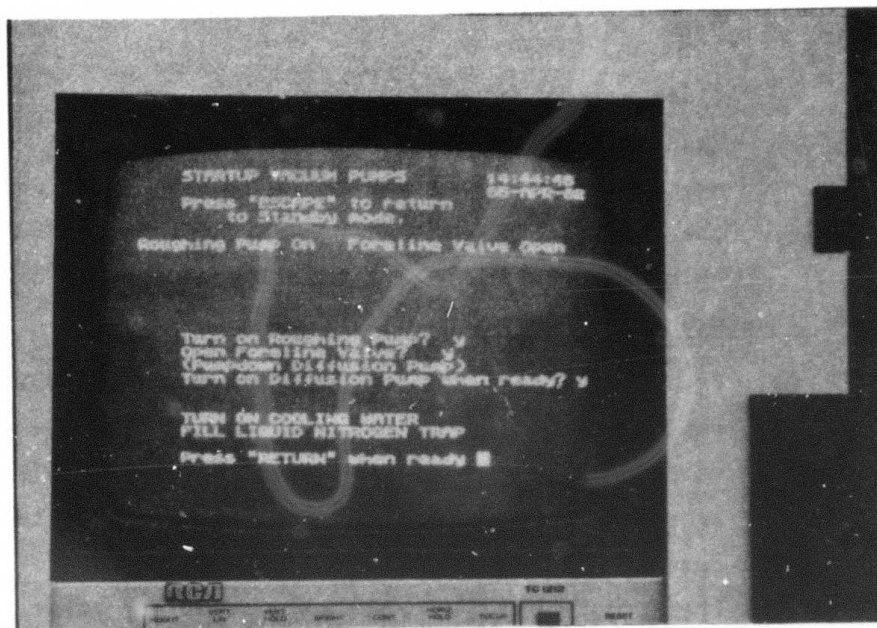
Pressing "Escape" will return to STANDBY MODE except where indicated.

2. 'Roughing Pump' and 'Foreline Valve' status indicators

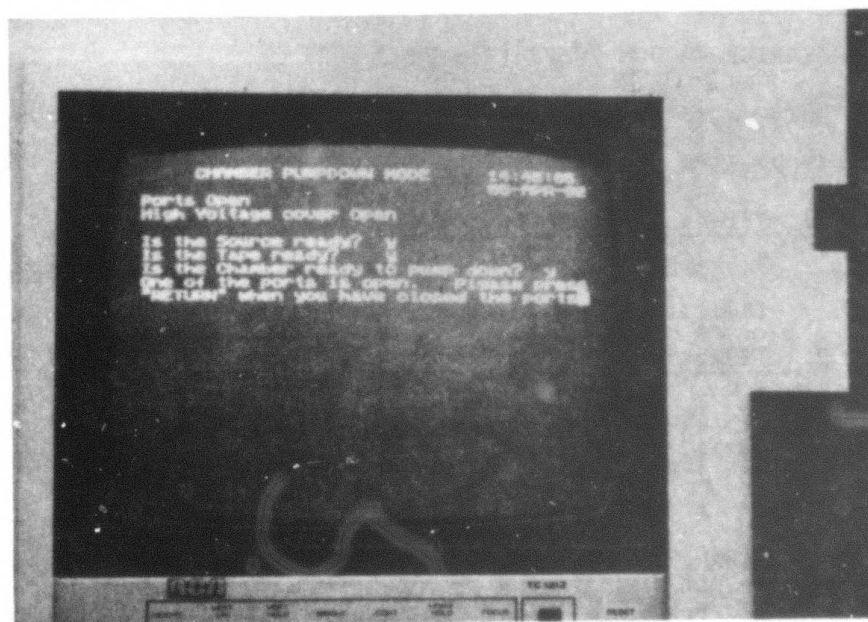
- A. 'On' or 'Off' will indicate present Roughing Pump status.
- B. 'Open' or 'Closed' will indicate present Foreline Valve status.

3. 'Turn on Roughing Pump?'

- A. Pressing "Y" or "Return" will start the pump.
- B. Pressing "N" will return to STANDBY MODE.



Startup Vacuum Pumps



Chamber Pumpdown Mode

Fig. 4-2 Video Screen Displays

4. 'Open Foreline Valve?' '(Pump down diffusion pump)'

- A. Pressing "Y" or "Return" will open the foreline valve.
- B. Pressing "N" will return to STANDBY MODE.

5. 'Turn on Diffusion Pump when ready?'

- A. Pressing "Y" will continue to next questions. Diffusion pump will not be turned on yet.
- B. Pressing "N" will skip to #7.

6. 'Turn on cooling water. Fill liquid nitrogen trap'

Press "Return" when ready

- A. Cooling water is required to operate the diffusion pump properly. See footnote (SVP1). An outlet (J116) has been provided at the rear of the Relay Module. See footnote (SVP2), to accommodate a solenoid valve (120 vac 1 amp max). This valve will then turn the water on and off at the appropriate times.
- B. Liquid Nitrogen is only required while the gate valve is open. See SVP3. Another opportunity to fill the LN2 trap will be provided just prior to opening the gate valve. This will allow for possible long time intervals between start-up and actual operation and reduce needless LN2 consumption.
- C. Pressing "Y" or "Return" will start the diffusion pump, turn on sequence and erase 'Press "Return" when ready'.
- D. Pressing "N" will repeat the statement.

7. 'Waiting for pressure < 50 millitorr'

This message will appear after "Y" or "Return" was pressed in response to 'Press "Return" when ready', or "N" was pressed in response to 'Turn on diffusion pump when ready?'.

- A. "Escape" will not function while this message is on the screen. The message will erase when the pressure is less than 50 millitorr, unless a leak occurs. See #8.

- B. If "Y" or "Return" is pressed after 'Turn on diffusion pump when ready?', the diffusion pump will turn on and the sequence will continue without further operator input, unless a leak occurs. The screen will return to STANDBY MODE when complete, unless "N" was answered to 'Turn on diffusion pump when ready?'. See #9.

8. 'Possible leak in Foreline - Try Again?'

- A. Diffusion pump was not turned on.
- B. This message only occurs if there is a leak or pumpdown was excessively slow.
- C. The cursor will return to the question, 'Open Foreline valve?'. Responses are the same as in #4 above.
- D. There is no limit on the number of attempts to pump down diffusion pump.

9. 'Turn on Diffusion Pump?'

This question is only asked if "N" was answered to the question, 'Turn on diffusion pump when ready?'.
A. If "Y" or "Return" is pressed, the procedure is the same as in #6, except the message, 'Waiting... < 50 millitorr', will not appear.

- B. If "N" is pressed, the diffusion pump will not be turned on. The screen will return to STANDBY MODE.

4.6 CHAMBER PUMPDOWN MODE (See Fig. 4-2)

"Escape" will return to STANDBY MODE except where indicated.

1. 'Ports Open' and 'High voltage cover Open'

- A. The appropriate message(s) will appear if either are open upon entry only. The messages are for information only.
- B. They will update if a change in status occurs by '... closed'.

2. 'Chamber already pumped down'

Press "Return" to return to Standby Mode'. Press "Return" if the procedures in this mode have already been completed.

3. 'Roughing Pump not on - Cannot continue'

'Press :Return" to return to Standby'. Pressing "Return", "Y", or "N" will return to STANDBY MODE.

4. 'Is the source ready?' and 'Is the Tape Ready?'

Press "Y" when ready. These messages are reminders only.

5. 'Is the chamber ready to pump down?'

Several checks are made automatically by the computer to insure the system is actually ready to continue.

A. "Y" will continue.

B. "N" will return to STANDBY MODE

6. 'One of the ports is open. Please press "Return" when you have closed the ports'

The port(s) must be closed before you may continue.

7. 'The high voltage cover is open. Do you wish to continue without high voltage?'

A. If "Y" is pressed, the high voltage short test will be skipped. High voltage may still be used in HEATUP AND OPERATE when the cover is replaced.

B. If "N" is pressed, the screen will return to STANDBY MODE.

8. 'Testing for high voltage short'

"Escape" is disabled during short test. If there is no short, the message will erase and the questions will continue.

NOTE

When the chamber is in the millitorr range, corona will allow enough current to flow to indicate a short. If this occurs, the test should be repeated by pressing "Escape" and re-entering PUMPDOWN CHAMBER MODE. Visual observation should be made, watching for any traces of corona.

9. 'A high voltage short exists. Do you wish to continue?'

- A. If "Y" is pressed, the test results will be ignored.
- B. If "N" is pressed, the screen will return to STANDBY MODE.

NOTE

If the diffusion pump is on, questions #10 through 13 will be skipped.

10. 'Rough pump the Chamber?'

- A. Pressing "Y" will open the roughing valve and put up the message, 'Pumping down Chamber', unless a port is open(ed). "Escape" is disabled while the 'Pumping down Chamber' message is being displayed. If the ports are open(ed), the pumping sequence will be cancelled. The procedure beginning at #6 must be repeated.
- B. Pressing "N" will return to STANDBY MODE.

11. 'Possible leak in vacuum chamber'. 'Do you wish to try again?'

- A. Pressing "Y" or "Return" will repeat the pumpdown sequence any number of times.
- B. Pressing "N" will return to STANDBY MODE.

12. 'Do you wish to leave the roughing valve open?'

- A. Pressing "Y" will leave roughing valve open and continue.
- B. Pressing "N" or "Escape" will close roughing valve and continue.

13. 'Chamber rough pumped. Press "RETURN" to return to Standby Mode'.

Pressing "Y", "N" or "Return" will return to STANDBY MODE.

NOTE

If the diffusion pump is off, this mode will not continue beyond this point.

14. 'Pumpdown chamber to High Vacuum?'

- A. Pressing "Y" will proceed to the next statement.
- B. Pressing "N" will return to STANDBY MODE.

15. 'FILL THE LIQUID NITROGEN TRAP' 'Press "Return" when ready'.

- A. Pressing "Return" will continue.
- B. Pressing "N" will return to STANDBY MODE.

16. 'Waiting for Diffusion Pump to heat up'.

'Pumping will continue at approximately HH:MM (H=hour; M=minute).

- A. This message will only be displayed if the diffusion pump has not been on long enough to heat up.
- B. If "Escape" is used here, the pumpdown sequence will be cancelled. The foreline valve will remain open and the diffusion pump on. The screen will return to STANDBY MODE.

17. 'Pumping down Chamber/Foreline'.

A. 'Chamber'

This message will be displayed while the roughing valve is open.

B. 'Foreline'

If the foreline pressure rises above 200 millitorr, the roughing valve will be closed and the foreline valve opened. When the foreline pressure drops below 50 millitorr, the foreline valve will close and the roughing valve will re-open. This may happen any number of times until the chamber pumpdown time is exceeded.

- C. If the port(s) are opened, the pumping sequence will be cancelled. The roughing valve will be closed, the foreline valve opened and the pumpdown procedure must be repeated from #6.

18. 'Possible leak in vacuum chamber'. 'Do you wish to try again?'.

The roughing valve has been closed and the foreline valve opened.

- A. Pressing "Y" or "Return" will proceed from #17.
- B. Pressing "N" will return to STANDBY MODE.

19. 'Possible leak in Foreline'. 'Diffusion Pump turned off'.

Press "RETURN" to return to standby.

- A. Roughing valve is closed. The foreline valve is open and the diffusion pump is off.
 - B. If "Y", "N", or "Return" are pressed, the screen will return to STANDBY MODE.
20. 'Chamber pumped down; press "RETURN" to return to Standby Mode'.
- A. The roughing valve is closed. The foreline and gate valves are open.
 - B. If "Y", "N", or "Return" are pressed, the screen will return to STANDBY MODE.

4.7 SOURCE HEATUP AND OPERATION (See Fig. 4-3)

- 1. 'Chamber not at high vacuum'.
Chamber pumpdown sequence was not completed. Skip to #24.
- 2. 'Waiting for Ion gauge to turn on'.
"Escape" is disabled while this message is being displayed.

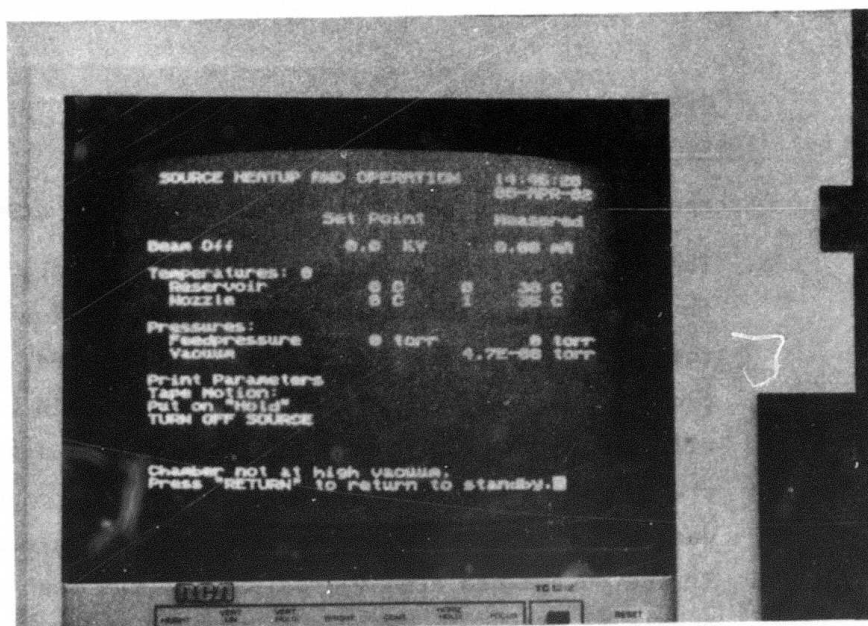
A delay of several seconds may occur while attempts are made to turn on the Ion Gauge.

- 3. 'Ion gauge not operating or possible leak. Unable to test for Nozzle blockage. Do you wish to continue?'.
 - A. Pressing "Y" will continue. The nozzle blockage test will be skipped. Skip to #9.
 - B. Pressing "N" will not continue.
- 4. 'Press "Return" to return to standby'.
Pressing "Y", "N", or "Return" will return to STANDBY MODE.

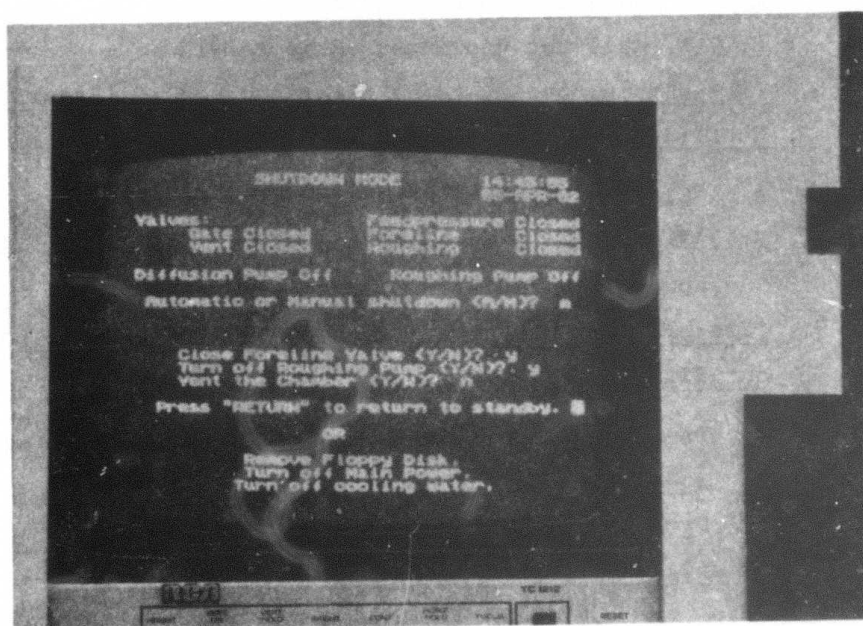
NOTE

Questions #5 through 8 will be skipped if "N" was answered to "Do you want to use feedpressure?" in SET PARAMETER MODE.

- 5. 'Should I test for Nozzle blockage?'.
 - A. Pressing "Y" will test for blockage.
 - B. Pressing "N" will not test for blockage. The feedpressure valve will be opened here (an audible solenoid valve sound



Source Heatup and Operation



Shutdown Mode

Fig. 4-3 Video Screen Displays

may be heard). Skip to #9.

6. 'Testing for Nozzle blockage'.

"Escape" will be disabled while this message is displayed.

- A. The computer performs this test by reading the ion gauge then increasing the feedpressure setpoint from 10 to 200 torr. When a pressure of 200 torr is reached, the feedpressure valve is opened (an audible solenoid valve sound will be heard). The ion gauge is then read again. If the second value gives a pressure more than one-half of a decade higher, the nozzle is assumed to be clear.
- B. If the charge has been melted before, the test may show the nozzle is blocked. This would be expected.
- C. If the pressure in the gas inlet line to the feedpressure manifold is less than 1000 torr or the gas inlet solenoid is either blocked or not functioning, the feedpressure may not reach the setpoint (200 torr) in the allotted time.

7. 'Feedpressure unable to reach setpoint'. 'Do you wish to continue?'.

- A. "Y" will continue as if everything is OK. Skip to #9.
- B. "N" will not continue. Skip to #13.

8. 'The nozzle appears to be clear/blocked'. 'Press "Return" to Continue'.

- A. 'Clear'. Pressing "Return" will continue.
- B. 'Blocked'. Pressing "Return" will continue. A chance to return to STANDBY MODE will be given later.

9. 'The high voltage cover is open. Do you wish to continue without High Voltage?'.

- A. Pressing "Y" will skip to #12. High voltage will be available later if the cover is replaced.
- B. Pressing "N" will skip to #13.

10. 'Testing for High Voltage short'.

The high voltage supply is turned on and set to 3.0kV. The voltage is set to 0.0 and the supply is turned off after the test. "Escape"

is disabled while this message is displayed.

NOTE

If no short exists, #11 will be skipped

11. 'A high voltage short exists. Do you wish to continue?'.
 - A. Pressing "Y" will proceed disregarding the short.
 - B. Pressing "N" will skip to #13.
12. 'Are you ready to heat the source?'.
 - A. Pressing "Y" will skip to #13.
 - B. Pressing "N" will give #13.
13. 'Press "Return" to return to standby'.
 - A. Pressing "Y" will close the feedpressure valve and return to STANDBY MODE.
 - B. Pressing "N" will re-ask #12.

There are three (3) additional features to the SOURCE HEATUP AND OPERATE display which are for use while trouble shooting the source heaters:

- A. Numbers ranging from 0-99 will appear between the Nozzle and Reservoir setpoints and measured temperatures. These numbers indicate the duty cycle of their corresponding heater.
- B. A single number ranging from 0-5 will appear to the right of the word, 'Temperatures'. The numbers indicate which heater transformer tap is presently being used. '0' indicates no tap.
- C. A dot will be displayed above one of the first six (6) letters of the word, 'Temperatures'. This dot indicates which heater state the μ PP is in. Its use is for software debugging and has little operator value.

Actual Source Heatup and Operation

Note: During printing, screen updating will be disabled. If characters are entered during this time, they may be lost.

Note: Initially the Nozzle and the Reservoir heaters will not be turned on until both setpoints are raised to at least 100°C. After the initial turn-on which enables the computer to determine if the thermocouples and the heaters are working properly, the setpoints may be changed independently.

During the actual operation of the powder processor, the operator will make entries directly into the setpoints. The following are commands used in this procedure:

Cursor Positioning:

- A. To move up one setpoint, press "^".
- B. To move down one setpoint, press "line feed".
- C. "Escape" will now move the cursor to 'Turn off Source'.

How to make an entry:

1. Once an entry is started, it must be completed before you will be allowed to continue.
2. To make an entry, place the cursor beside the desired setpoint.
3. Enter the desired value (omit units. e.g., kV,C,torr).
4. If you make a mistake, use "Delete", and re-enter the correct value.
5. Press "Return" when the entry is correct.
6. If a value is entered that is disallowed, over/under range, the computer will replace it with the maximum/minimum value and display a message indicating an error. If you wish to use this value, press "Return". If not, use the delete key and enter your new choice.

14. 'Print Parameters'.

- A. "Y" will cause a printing of the setpoints and measured values. Automatic printing will occur whenever the operator makes a change or when the measured inputs change more than a program determined amount.
- B. "N" will disable automatic printing of measured input changes. Entries made by the operator will still cause printing to occur.
- C. "Return" will print the present values once and will not affect automatic printing status.

15. 'Tape Motion'.

- A. "N" will cancel present tape motion and close the shutter.
- B. "Return" and "Y" will initiate tape motion procedure, cancelling present motion, if any, and closing the shutter.

16. 'Tape Speed: (Slow, Medium, Fast or None)'.

The tape will advance rapidly to a clean area, then begin moving at the desired speed. The shutter will open and close automatically at the beginning and end of the sampling.

- A. "S" slow speed, run one second, stop three.
- B. "M" medium speed, run one second, stop one second.
- C. "F" fast speed, run continuously. Fast speed is approximately $\frac{1}{2}$ inch/minute.
- D. "N" stopped, does not move after initial rapid advancement.

17. 'Sample Time (in Minutes)'.

Enter desired sample time in minutes and tenths. e.g., MMM.T, up to 300 minutes in .1 minute increments.

18. 'Tape Motion [Fast, Medium, Slow, Stopped]'.

'Sample will be done at: HH:MM:SS.

The time shown is the time the sample will be complete. A printing will occur automatically at the beginning and end of the sample to record the conditions for later reference. Indication will be made by

the printer to indicate the tape is sampling.

19. 'Put on Hold'.

- A. "Y" and "Return" will cause the nozzle and reservoir temperatures to be set to 200°C, and set the feedpressure to 10 torr. This option was provided to allow the operator a quick way to set up conditions enabling him to attend to other matters not relating to the μ PP (phone calls, etc.). Tape motion and printing status are unaffected.
- B. "N" is the same as "Line Feed" at this point.

20. '"ON HOLD" - Press RETURN to continue'.

- A. "Return" will place the cursor at the Reservoir Temperature Setpoint.
- B. "Escape" will place the cursor at 'Turn off Source'.

21. 'Turn off Source'.

- A. "N" will place the cursor at the source voltage setpoint.
- B. "Y" will initiate a computer controlled turn-off sequence. The present values will be printed once automatically, printing status will be unaffected (during a typical source cool down, several pages of values will be printed if the Print Parameter status is "Y").

NOTE

"Escape" will not cause a cancellation of the controlled turn-off sequence and any other selected options. The cursor will be placed beside the Reservoir setpoint. The Reservoir and Nozzle setpoint will be set to their present temperature. Operator input will then be restored. If Turn-Off Source was selected and the printing status is not the desired status, using "Escape" is a method of correcting this oversight.

22. 'Perform complete System Shutdown?'.

(In addition to turning off the source.)

- A. "N" will turn off the source only. When the Nozzle and

Reservoir temperatures are below 100°C, skip to #24.

- B. "Y" will cause the computer to cool down the source to 100°C, then proceed to SHUTDOWN. In SHUTDOWN, the automatic procedure will be performed up to turning off the roughing pump.

23. 'Vent chamber when done?'.

- A. "Y" will vent the chamber after the gate valve has been closed.
- B. "N" will not vent the chamber. Another chance will be given in SHUTDOWN.

'Turn Off Source' Procedure

- a. The heater transformer tap will be set to tap 1.
- b. The Feedpressure will be set to 10 torr.
- c. The Reservoir and Nozzle setpoints will be set to zero.
- d. The Parameters will be printed once to record the time.

When the Reservoir temperature is less than 300°C or after 120 minutes, TURN OFF SOURCE will continue.

- e. The Source Voltage will be set to zero.
- f. The Feedpressure valve will be closed.
- g. The Tape Drive will be stopped if in motion, and the shutter closed.
- h. The Parameters will be printed once for the record.
- i. If complete system shutdown was selected, the diffusion pump heater will be turned off at this point.

When the Reservoir and Nozzle temperatures drop below 100°C or after sixty (60) minutes TURN OFF SOURCE will continue.

- j. "Escape" will not longer exit from TURN OFF SOURCE.
- k. The heater transformer will be turned off.
- l. Updating of the Parameter values will discontinue.
- m. Parameters will print once.
- n. If Complete System Shutdown was selected, skip to SHUTDOWN #1.

24. 'Press "RETURN" to return to standby'.

- A. "Y" will return to STANDBY MODE.
- B. "N" will skip to #1 of this section.

4.8 SHUTDOWN MODE (See Fig. 4-3)

Note: If automatic shutdown was not selected in HEATUP AND OPERATE, Skip #1.

Note: Generally automatic should be used if a complete system shutdown is to be performed. Manual is useful to close the gate valve and vent the chamber, leaving the pumping system ready to resume another run.

Note: "Escape" will cancel any unexecuted procedures if used to exit, e.g., during the time the 'Waiting for Diffusion pump to cool down'.

1. 'Automatic Shutdown'.

- A. This message will be displayed if complete automatic system shutdown performance was requested in HEATUP AND OPERATION.
- B. The chamber will be vented automatically when the system is ready, if requested from HEATUP AND OPERATE. If chamber venting was not requested from HEATUP AND OPERATE, a second opportunity will be offered when the diffusion pump has cooled down.

NOTE

If complete automatic vacuum system shutdown was requested from SOURCE HEATUP AND OPERATE, SKIP TO #4.

2. 'Automatic or Manual shutdown (A/M)?'.

- A. "A" Automatic will perform a vacuum system shutdown with only a vent chamber question.
- B. "M" Manual allows the operator to turn off selected portions of the vacuum system.

3. 'Do you wish to vent the chamber?'.

- A. "Y" will vent the chamber after the gate valve is closed.
- B. "N" will not vent the chamber. Another opportunity will be given at the end of the automatic vacuum system shutdown sequence.

4. 'Waiting for Diffusion Pump to Cool off'. 'Do not turn water off until HH:MM'.

- A. This message is to remind the operator that the diffusion pump has not cooled down enough to turn off the cooling water.
- B. This message will be erased when it is safe to continue.
- C. If "Escape" is used to exit from this point, remainder of automatic vacuum system shutdown will be cancelled.

5. 'OK to turn off cooling water'.

Diffusion pump cooling water solenoid valve will be closed automatically at this point.

6. 'Do you wish to vent the chamber?'.

This question will not be asked if "Y" was answered to #3.

- A. "Y" will vent the chamber.
- B. "N" will not vent the chamber.

7. 'Press "RETURN" to return to standby'.

'OR'

'Remove Floppy Disc'.

'Turn off Main Power'.

'Turn off Cooling Water'.

MANUAL

8. 'Close Roughing Valve (Y/N)?'.

- A. "Y" will close the roughing valve, skip to #15.
- B. "N" will not close the valve, skip to #17.

9. 'Close Gate Valve (Y/N)?'.
- A. "Y" will close gate valve, and turn off the vacuum Ion Gauge.
 - B. "N" will not close gate valve and return to Standby will be the only offered option. Skip to #17.
10. 'Vent the Chamber (Y/N)?'.
- A. "Y" will vent the chamber.
 - B. "N" will not vent the chamber.
11. 'Turn off the Diffusion Pump (Y/N)?'.
- A. "Y" will turn off the diffusion pump, and start a 45 minute cooldown timer before allowing the foreline valve to be closed or the roughing pump to be turned off.
 - B. "N" will not turn off the diffusion pump and will skip to #16.
12. 'Waiting for Diffusion Pump to cool off'.
- 'Do not turn water off until HH:MM'.
- A. No operator input is required until the time shown. Using "Escape" to exit from this waiting period will not affect system shutdown as no other procedures have been selected.
 - B. An option at this point would be to press "Escape", activate SHUTDOWN again from STANDBY MODE and proceed using automatic vacuum system shutdown to free operator from any further need to respond to messages.
13. 'OK to turn off cooling water'.
- Diffusion pump cooling water may be turned off. If solenoid valve is installed, water will be turned off automatically at this point.
14. 'Close Foreline Valve (Y/N)?'.
- A. "Y" will turn off roughing pump.
 - B. "N" will not close the foreline valve, skip to #15.

15. 'Turn off Roughing Pump (Y/N)?'.
A. "Y" will turn off roughing pump.
B. "N" will not turn off roughing pump.
16. 'Vent the chamber (Y/N)?'.
A. "Y" will vent the chamber.
B. "N" will not vent the chamber.
17. 'Press "RETURN" to return to standby'.
Press "Return"
18. 'Press "RETURN" to return to standby'.
'OR'
 'Remove Floppy Disc'
 'Turn of Main Power'
 'Turn off Cooling Water'
- Turn off water, do one of the above.

FOOTNOTES

- SM1 The thermal cutout switch at the bottom backside of the diffusion pump must be reset by pressing small round metal button. The diffusion pump temperature must be below the cutout point before button will remain depressed.
- SPM1 Testing for nozzle blockage and applying feedpressure will not be possible if "N" is pressed at the question, 'Do you want to use feedpressure?'. If "N" is pressed, the alternate source clamp should be used to avoid a glow discharge in the feedpressure line. The "N" option has been provided for cases where the feedpressure necessary to prevent a flow discharge in the feedpressure line would be excessive for the material being processed, such as a wetting material.
- SVP1 Cooling water must be circulated through the diffusion pump whenever the heater is on and approximately 45 min. after it has been turned off. Diffusion pump cooling water inlet is marked "C", outlet is marked "D". Water ports marked "A" and "B" are for vacuum chamber cooling water. Chamber cooling water flow may be in either direction.
- SVP2 The Relay Module is the power distribution and control point for the μ PP. To access J116, remove the left rear panel of the μ PP. The Relay Module is mounted uppermost in the internal instrument rack.

SVP3 Liquid Nitrogen (LN2) is only required while the gate valve is open. If extensive time may pass between starting up the diffusion pump and actual chamber pumpdown, LN2 may be conserved by not adding LN2 at this point in the procedure. A second message to add LN2 will occur prior to chamber pumpdown.

SECTION 5.
MAINTENANCE

5.1 REMOVAL OF TOP FLANGE FROM VACUUM CHAMBER

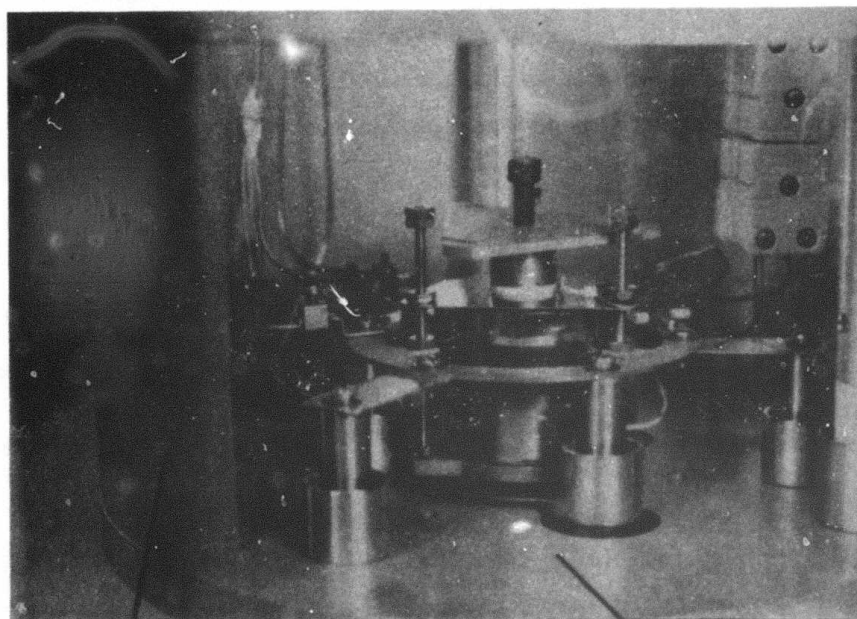
This part describes the removal of the top flange (with source assembled) from the vacuum chamber for source maintenance or other operations. Normally the reservoir assembly can be loaded or unloaded from the Micro-Particle source without removing the top flange as described in Section 3.2 and 3.1. Among the operations which may require removal of the entire source assembly from the vacuum chamber include:

- a. Replacement of nozzle and/or reservoir heater.
 - b. Clean and/or replace various source parts as required.
 - c. Replacement of nozzle and/or reservoir thermocouple.
1. Vent chamber as instructed by Shutdown Mode.
 2. Open source vacuum port by loosening four (4) wing nuts on door latches being careful not to dislodge O-ring.
 3. Reaching through the source vacuum port, gently lift door from cylindrical heat shield to disengage clips and remove through port opening. (See Fig. 5-1 for a view of the source after the door has been removed.)

NOTE

To prevent misalignment of Micro-Particle source parts when removing top flange, check to make sure there is a reservoir assembly in the source.

4. Standing on a secure ladder or staircase, remove the five (5) bolts which fasten the high voltage cover to the high voltage tower and vacuum chamber.
5. Lift off the high voltage cover and store in safe location.



Cylindrical Heat Shield

Source Support Plate

Fig. 5-1 Microparticle Source as seen through door of cylindrical heat shield.

6. Remove four (4) screws holding cover lid on EMI shield box. Lift off lid.
7. Loosen hose clamp on tygon feedpressure line located at TF4. (Refer to Fig. 5-2 and Table 5-1 for feedthrough locations.) Slide feedline through metal turret extending from EMI shield box. (Turret denoted by A in Fig. 5-3.)
8. Loosen (do not remove) three (3) screws located at the top of feedthroughs TF1, TF2 and TF3 and disengage heater leads.
9. Unplug thermocouple (2) connectors, located on feedthrough TF6.
10. Disengage the three (3) heater cable connectors denoted by B, C, and D in Fig. 5-3, located at the rear of the EMI shield box.

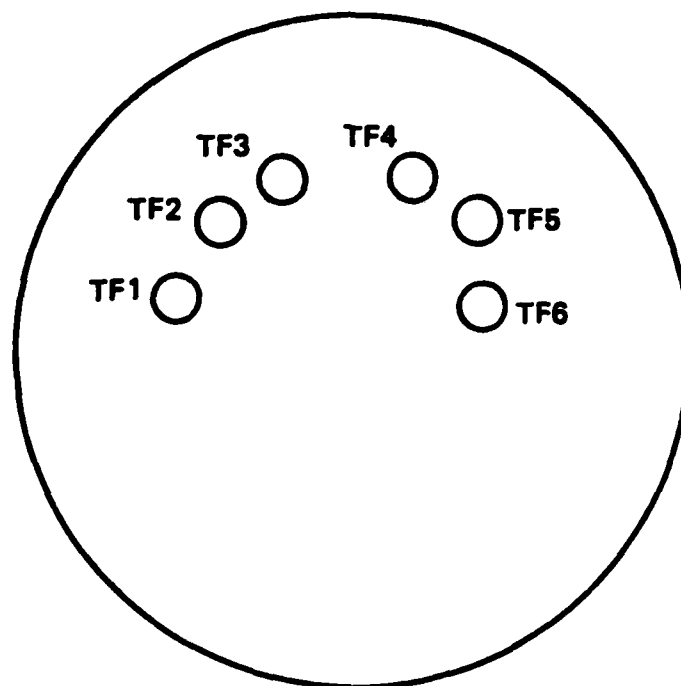
NOTE

Carefully place cables aside to prevent them from slipping through opening in the high voltage tower.

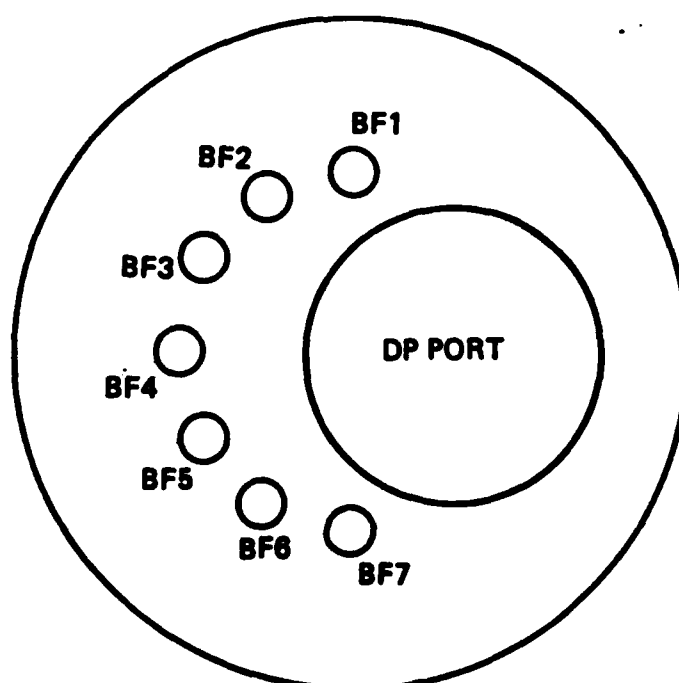
11. Remove four (4) screws holding thermocouple connector plate (designated by E in Fig. 5-3) on EMI shield box.
12. Remove plate assembly without disengaging cable from connector.
13. Remove four (4) mounting screws located at the base of the EMI shield box which fastens the box to the top flange.
14. Unscrew the three (3) aluminum top flange lift-off supports and remove.
15. Unscrew the two (2) rear standoffs mounted on top of the high voltage tower and remove.
16. Lift off the high voltage cover gasket plate and rotate clockwise, allowing the plate to rest on top of the computer console.
17. Replace the previously removed three (3) top flange lift-off supports.
18. Loosen and remove all 3/4" top flange bolts.

CAUTION

Because of the heavy weight, removal of the top flange from the vacuum chamber should be carried out with the aid of two (2) persons.



TOP PLATE (TOP VIEW)

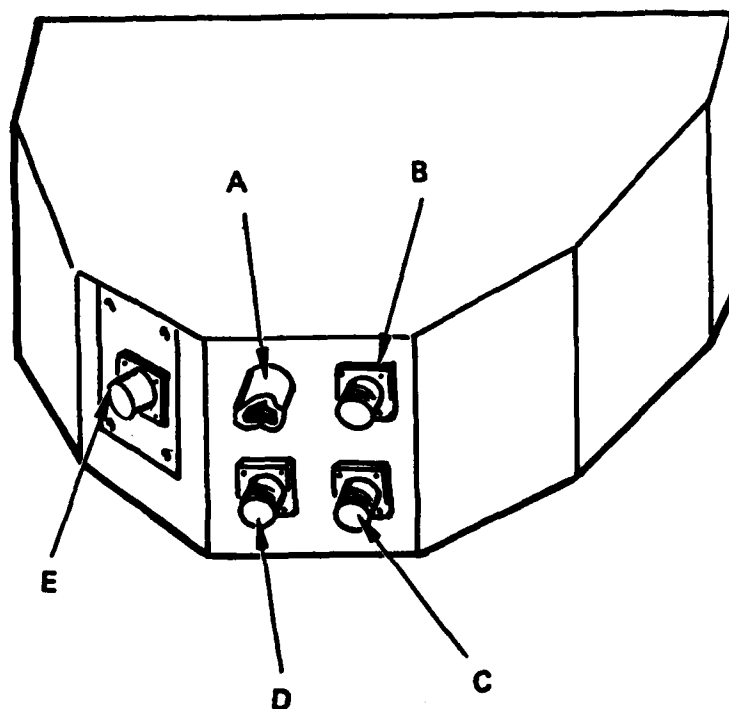


BASE PLATE (TOP VIEW)

Fig. 5-2 Top and Base Plate Feedthrough Locations

TABLE 5-1.
TOP AND BASE PLATE FEEDTHROUGH DESIGNATIONS

FEEDTHROUGH DESIGNATION	DESCRIPTION
Top Plate	
TF1	Heater Return
TF2	Reservoir Heater
TF3	Nozzle Heater
TF4	Feedpressure Line
TF5	Unassigned
TF6	Thermocouple (2)
Base Plate	
BF1	Tape Drive Stepper Motor
BF2	Tape Drive Shutter Solenoid
BF3	Unassigned
BF4	Unassigned
BF5	Unassigned
BF6	Unassigned
BF7	Unassigned



- A. FEEDPRESSURE LINE
- B. NOZZLE HEATER
- C. HIGH VOLTAGE/HEATER COMMON
- D. RESERVOIR HEATER
- E. THERMOCOUPLE (2)

Fig. 5-3 EMI Shield Box for Source Flange Insulators

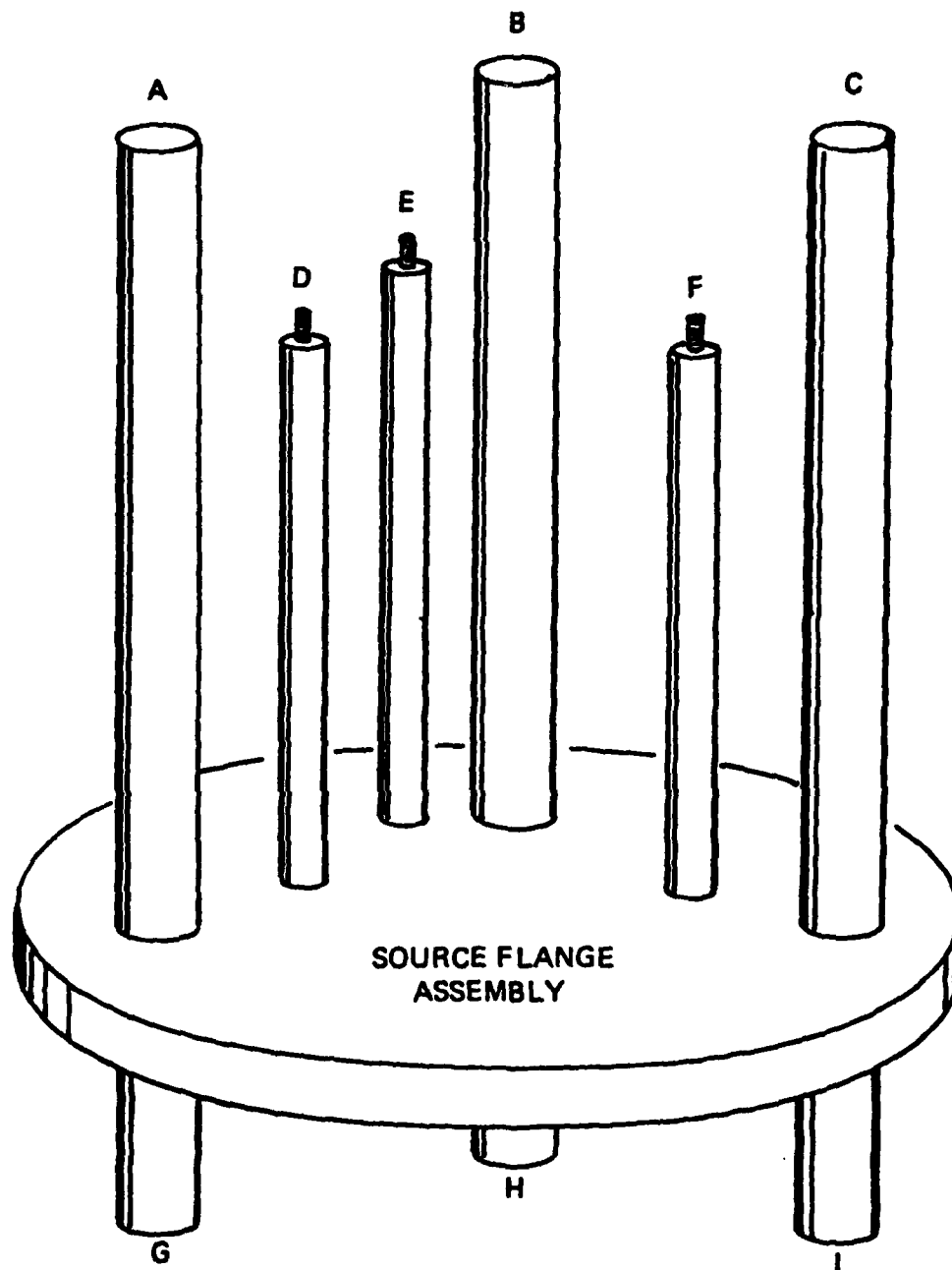
19. Firmly grasp two (2) of the top flange lift-off supports and lift flange straight up until all fixtures mounted on flange are clear of the chamber.
20. Carefully transfer the flange to the second person standing below for placement on nearby workbench.
21. Rest the top flange assembly on the three (3) aluminum lift-off supports (micro-particle source facing upwards) on workbench.

The top flange assembly is now in the starting position to initiate micro-particle source assembly. Instructions for source disassembly and assembly refer to various source flange standoffs and supports. Fig. 5-4 is included to identify the various standoffs referred to in these instructions.

5.2 DISASSEMBLY OF MICRO-PARTICLE SOURCE

Refer to instruction #21 of Section 5.1 and the statement which follows this instruction.

1. Remove the three (3) nuts which mount the cylindrical heat shield to the source assembly.
2. Disengage the extractor grounding wire from the screw stud.
3. Lift off the cylindrical heat shield.
4. Replace the three (3) previously removed nuts on the source standoff screw studs and fasten.
5. Gently lift up the source flange assembly and unscrew the three (3) top flange lift-off supports approximately halfway -- but do not remove.
6. Screw three (3) aluminum bench standoffs (12" long) provided into the partially vacated screw threads which hold the top flange lift-off supports.
7. Invert the entire source flange assembly and rest on the newly installed bench standoffs.
8. Loosen, but do not remove small allen screws (4) on stainless



A, B, C - BENCH STANDOFFS
D, E, F - SOURCE STANDOFFS
G, H, I - TOP FLANGE LIFT-OFF SUPPORTS

Fig. 5-4 Standoffs and Supports Referred to in Micro-Particle
Source Assembly and Disassembly Instructions

clamps which connect the source heater leads to the flange leads.

9. Slide stainless clamps part way onto copper flange leads for storage.
10. Loosen screw (do not remove) on source support ring and remove high voltage common/heater return lead.
11. Carefully disengage two (2) thermocouple connectors while supporting the insulator assembly on the top of the flange to prevent stressing.
12. Loosen thumb screw on the reservoir clamp plate and remove plate.
13. While supporting the 9 $\frac{1}{2}$ " Dia. source support plate, remove the three (3) nuts holding the plate to the source standoff studs.
14. After verifying that all source leads are disconnected from flange leads, carefully lower the support plate to disengage from source stand-off studs and remove assembly.
15. Using a small open-end wrench, loosen and remove the three (3) top nuts from the double set on the threaded support rods while holding the lower nuts of the set with a second open-end wrench. (See Fig. 5-5 for location of the double nut set.)
16. Remove the lower nuts of the double set.
17. Remove the bottom set of three (3) nuts on the threaded support rods holding the Tantalum heat shield cover.
18. Remove the reservoir thermocouple insulator clamp (refer to Fig. 5-5 for part location).
19. Carefully lift off the reservoir thermocouple assembly.
20. Remove the Tantalum heat shield cover from the threaded support rods.
21. Loosen the reservoir heater lead insulator clamp screw and remove the two (2) ceramic insulator tubes. (See Fig. 5-6.)

CAUTION

Do not bend heater leads to prevent breakage.

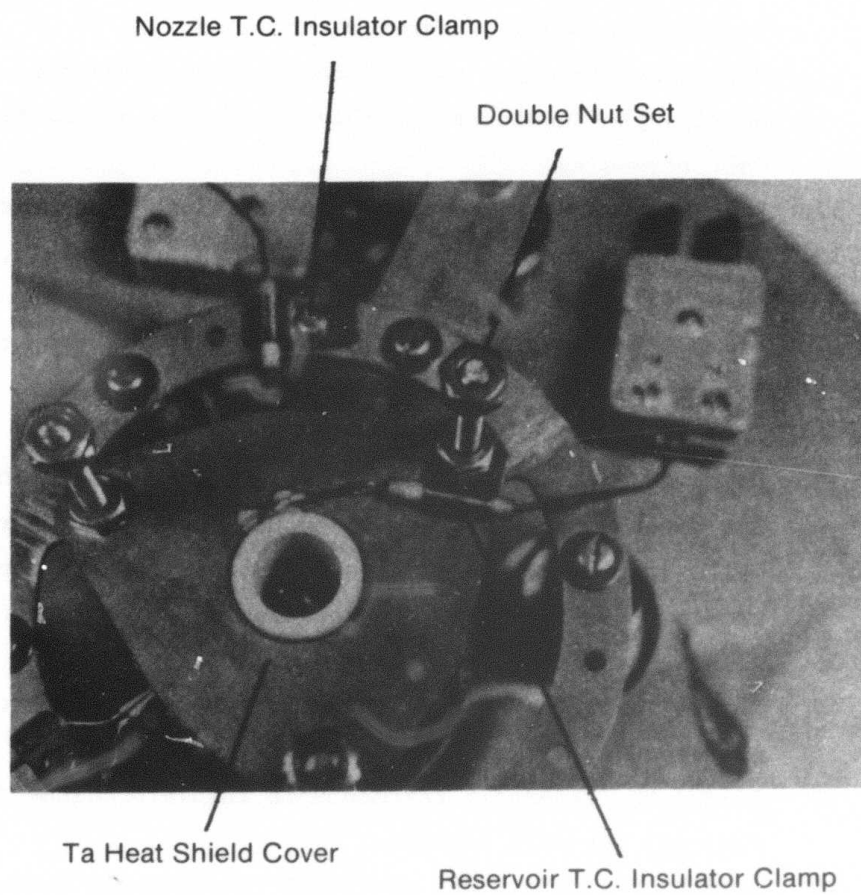


Fig. 5-5 Location of Source Parts Referred to in Disassembly Instructions

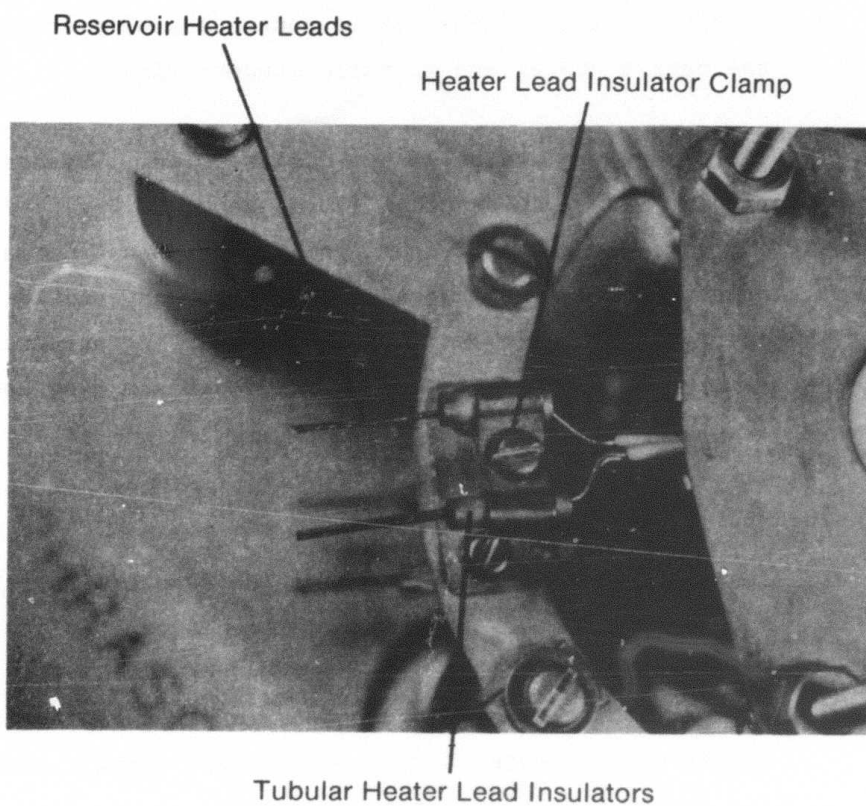


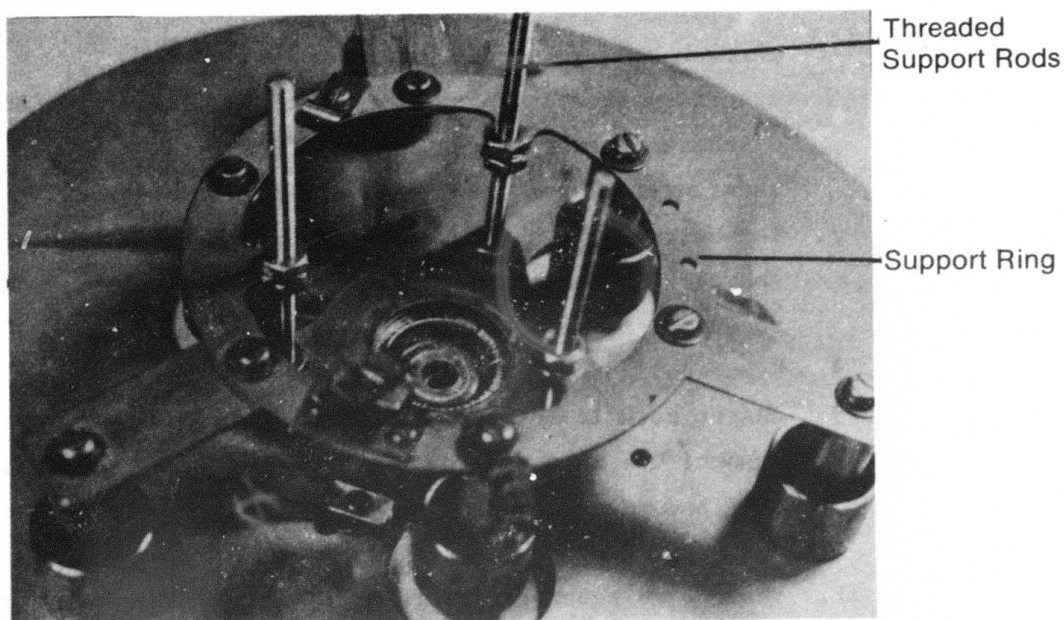
Fig. 5-6 Reservoir Heat Lead Assembly

22. Carefully lift off the micro-particle source oven (including the heat shield structure and reservoir heater assembly).
23. Remove the ceramic reservoir assembly.
24. Loosen screw on nozzle thermocouple insulator clamp and remove thermocouple assembly. (See Fig. 5-5.)
25. Loosen the nozzle heater lead insulator clamp screw and remove the two (2) ceramic insulator tubes.
26. Carefully lift off the nozzle heater assembly consisting of the nozzle heater and graphite support ring.

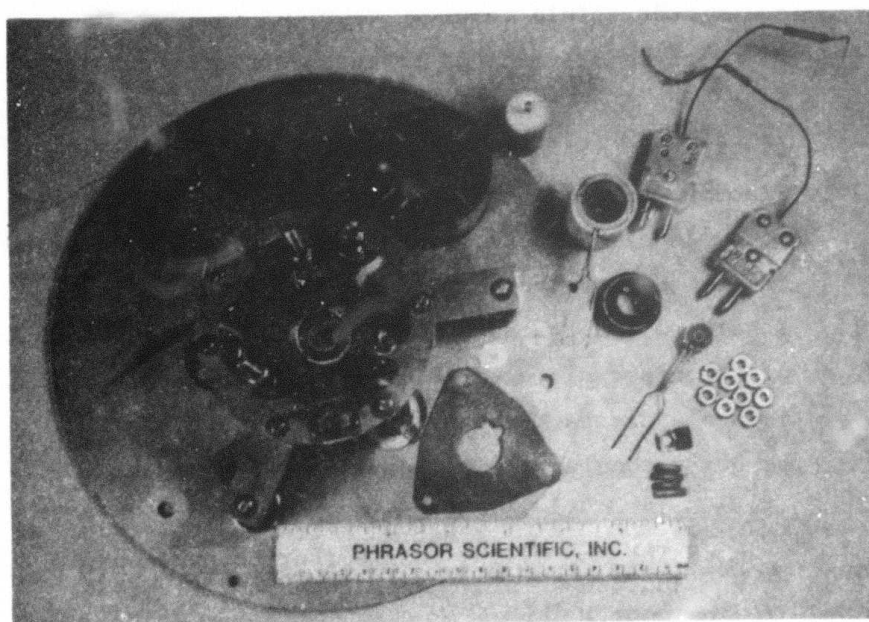
The micro-particle source is now disassembled. The source support structure remaining after instruction #26 is shown in Fig. 5-7. Further disassembly of source support structure components is not normally required unless molten metal leaks have damaged the Tantalum cap shield or other parts. The configuration as shown is also the starting structure for source re-assembly. Disassembled source components are also shown in Fig. 5-7.

5.3 ASSEMBLY OF MICRO-PARTICLE SOURCE

1. Inspect the bottom boron nitride (BN) washer insulator. If broken or darkened by metal vapor deposits, replace with new washer supplied with source maintenance kit.
2. If nozzle heater is burned out or requires replacement, disengage nozzle heater body from graphite support ring. Insert new nozzle heater assembly.
3. Remount nozzle heater and graphite support ring being careful to align nozzle heater leads with nozzle heater lead insulator clamp.
4. Inspect the upper nozzle heater BN washer insulator. If broken or darkened by metal vapor deposits, replace with new washer supplied with the source maintenance kit.
5. Insert alignment rod (1/8" O.D. stainless steel rod 3-4" long with tapered end) provided with the source maintenance kit



Source Structure Remaining After Disassembly



Disassembled Source Components

Fig. 5-7 Disassembled Configuration of Micro-Particle Source

through hole in nozzle heater body.

6. If reservoir heater is burned out or requires replacement, disengage reservoir heater body from reservoir oven shield structure. Insert new reservoir heater assembly.
7. Remount assembled reservoir oven and heater structure on source being careful to align reservoir heater leads with reservoir heater lead insulator clamp.
8. Remove alignment rod.
9. Insert ceramic reservoir assembly into reservoir oven structure.
10. Remount the Tantalum heat shield cover over threaded support rods and lower until it makes contact with the top of reservoir oven structure.
11. Insert the reservoir thermocouple through the slot provided in the side of the BN reservoir heater core. The thermocouple should insert a distance of one-half to three-quarter the length of the reservoir oven structure.
12. Place reservoir thermocouple clamp over threaded support rod as seen in Fig. 5-5 and lower until clamp rests on top of Tantalum heat shield cover.
13. Place three (3) nuts on threaded support rods and screw to the bottom to lock the Tantalum heat shield in position (finger tight).
14. Place reservoir thermocouple ceramic insulator tube under thermocouple clamp. Use one of the previously installed nuts to fasten the reservoir thermocouple clamp.
15. Insert nozzle thermocouple through hole located at the base of the reservoir oven shield structure making sure that thermocouple passes through the hole in the ceramic insert on the graphite nozzle heater ring.

CAUTION

The end of the nozzle thermocouple should not physically contact the nozzle heater body.

16. Place nozzle thermocouple ceramic insulator tube under insulator clamp located on source support ring as seen in Fig. 5-5 and secure by tightening clamp screw.
17. Place new GRAFOIL gasket on reservoir assembly sealing surface.

The following few instructions refer to mounting a special reservoir clamp plate assembly that is normally used with the micro-particle source for alloys which wet source parts and do not require feedpressure. Once the source is assembled, the plate is replaced with the operating reservoir clamp plate and feedpressure inlet cap used for nonwetting operations that require feedpressure. The special clamp plate is used only to secure source components during the remaining assembly steps.

18. Set the special reservoir clamp plate onto the ceramic reservoir assembly.
19. Rotate the clamp assembly to engage slots with the threaded support rods.
20. Screw three (3) nuts on threaded support rods.
21. Using a small 6" machinist rule, measure the distance from the top of the bottom source support plate (molybdenum) to the bottom surface of the reservoir clamp plate. (This vertical distance should be measured along side of the threaded support posts and as near as possible.) This distance should be 2-5/8" -- if not, adjust until all distances are equal.
22. Screw a second set of three (3) nuts onto the threaded support rods.
23. While pressing down on the reservoir clamp plate, loosen thumb screw until clamp plate lowers clear from nuts.
24. Making sure that the bottom set of nuts above clamp plate do not rotate by holding with open-end wrench, lock the top nut set against the bottom nut, using a second open-end wrench.
25. Tighten the thumb screw on the reservoir clamp plate until clamp plate rises and makes contact with the bottom nut set.
26. Place ceramic heater lead insulators over reservoir heater

leads and insert into insulator clamp. Tighten clamp screw.

NOTE

Avoid stressing heater leads to prevent damage.

27. Place ceramic heater lead insulators over nozzle heater leads and insert into nozzle heater clamp. Tighten clamp screw.
28. Firmly holding the 9½" Dia. source support plate, lift source assembly for mounting on source standoffs.
29. Engage support plate on source standoff studs making sure that source heater leads are on the same side and aligned with connecting flange leads.
30. Place three (3) nuts on standoff studs and fasten source support plate by tightening nuts.
31. Insert high voltage common/heater return flange lead into clamp located on source support ring and tighten screw.
32. Insert reservoir heater leads into stainless steel clamps previously stored on copper flange leads and tighten allen screws.
33. Insert nozzle heater leads into stainless steel clamps provided on copper flange leads and tighten allen screws.
34. Plug thermocouple connectors into mating connectors extending from flange feedthrough.

NOTE

Make sure that nozzle and reservoir thermocouple connectors are plugged into its proper mating connector. Connectors are marked "N" for nozzle and "R" for reservoir.

35. Loosen thumb screw on special reservoir clamp plate and rotate to disengage from threaded support posts. Remove the clamp plate.
36. Mount feedpressure inlet cap on flexible feedpressure line onto the ceramic reservoir sealing surface.
37. Mount the operating reservoir clamp plate and thumb screw

assembly onto the source. Rotate to engage plate on threaded support posts.

38. Tighten thumb screw with fingers to make seal.
39. Invert the entire source flange assembly and rest on the three (3) top flange lift-off supports.
40. Remove the three (3) nuts from the source standoff studs.
41. Replace cylindrical heat shield cover with door opening directly opposite the flange feedthroughs.
42. Engage extractor grounding wire on nearest source standoff stud.
43. Replace flat and split washers on all three (3) source standoff studs.
44. Replace nuts on source standoff studs and tighten.

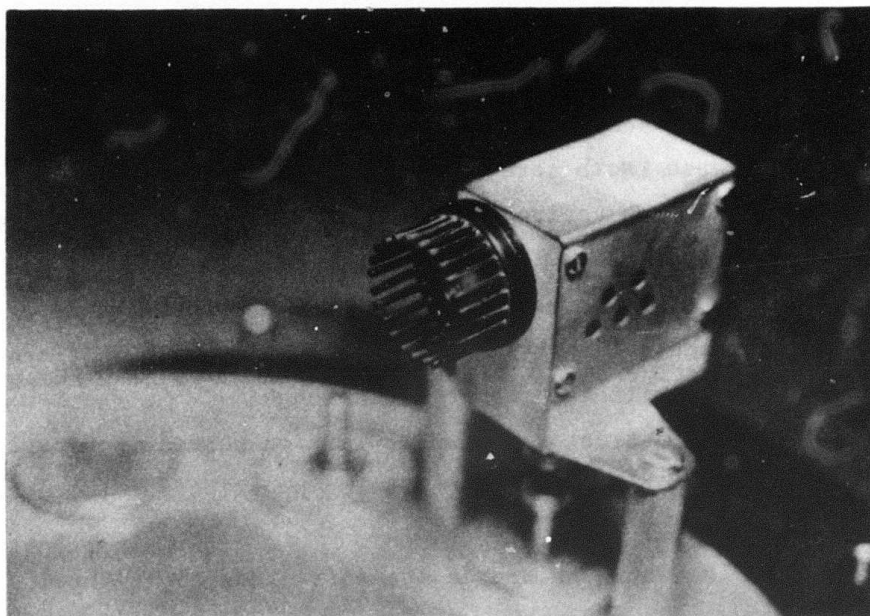
The micro-particle source is now assembled. After removing three (3) bench standoffs, the source flange assembly is now ready for mounting onto the vacuum chamber.

5.4 REMOVAL OF THE TAPE DRIVE ASSEMBLY FROM VACUUM CHAMBER

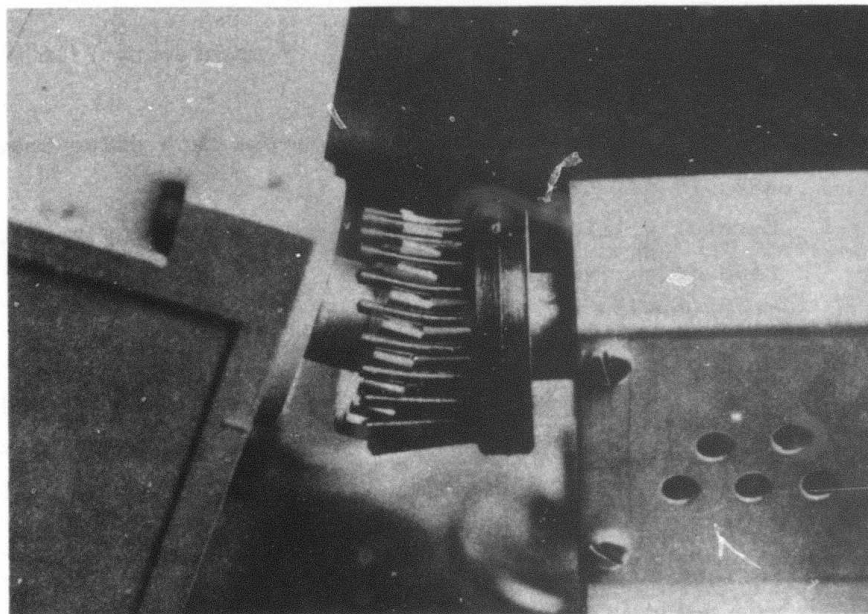
1. Remove tape drive shutter (12) by unscrewing 7/16" bolt located at shutter pivot point.
2. Loosen and remove allen screw on the tape drive assembly mounting bracket (7).
3. Carefully lift tape drive assembly and gently remove through tape drive vacuum port.

Installation of Tape Drive Assembly

1. Before installing tape drive assembly, clean base plate flange and tape drive components by removing all particulates.
2. Insert tape drive assembly through lower vacuum port.
3. Tilt the tape drive assembly toward the rear of the vacuum chamber to initiate the engagement of the flexible pins on the Flex-Thane coupling with the capstan tooth sprocket (refer to Fig. 5-8).



Tape Drive Assembly Flex-thane Coupling



Position for Engaging Flexible Coupling

Fig. 5-8 Flexible Coupling Assembly

4. Level tape drive assembly and gently press assembly all the way to the rear.
5. Verify that all the flexible pins have properly engaged the tooth sprocket as shown in Fig. 5-8.

NOTE

Misaligned or unengaged pins may be inserted into place using small, blunt instrument.

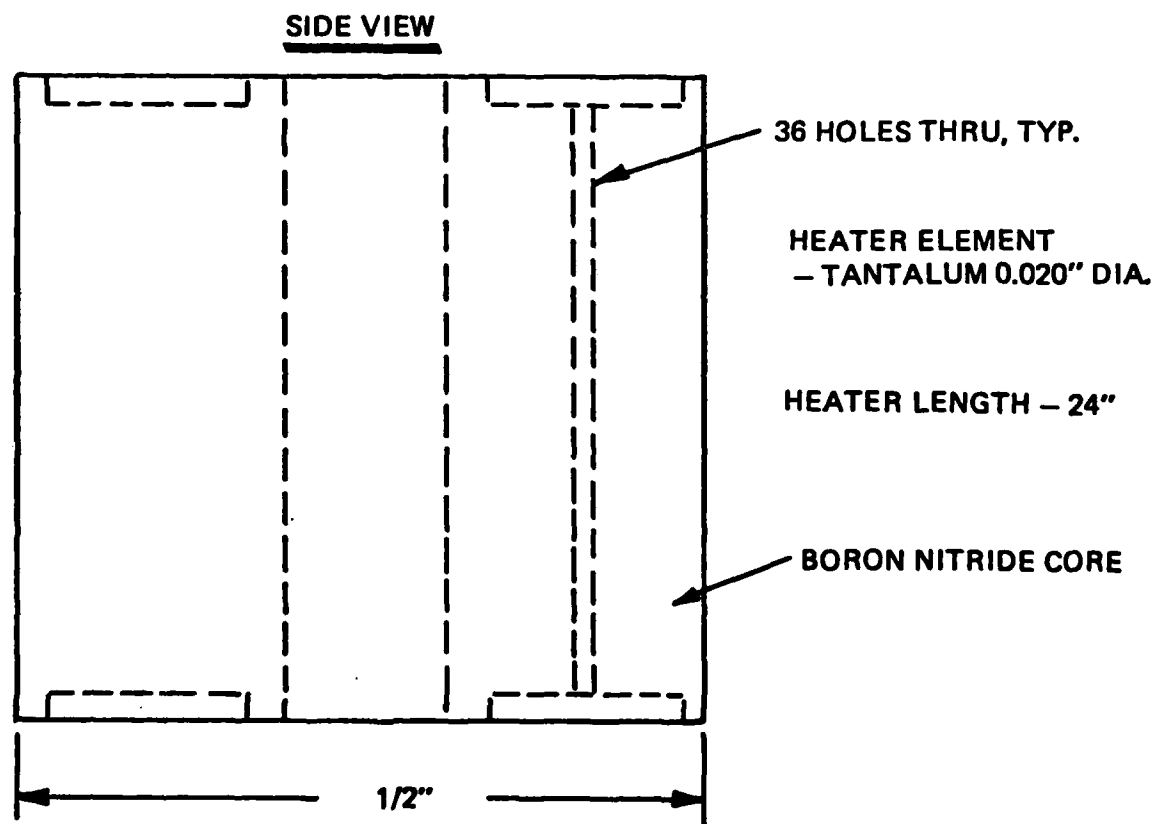
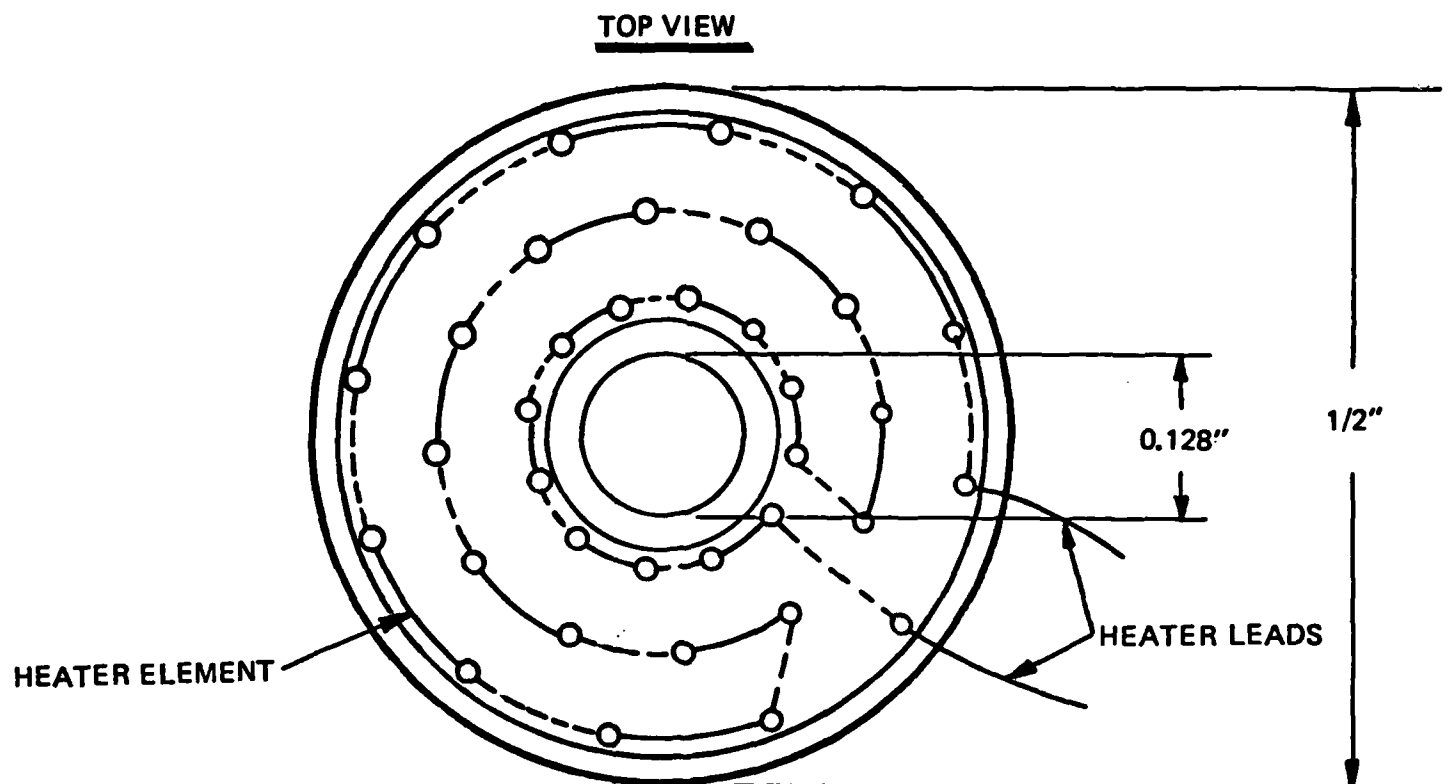
6. Reinsert allen screw into tape drive assembly mounting bracket (7) and tighten.
7. Replace tape drive shutter (12) and tighten 7/16" bolt at pivot mount -- centering the shutter over sample tape aperture.
8. Remount tape drive aperture plate (11) and fasten, using two (2) thumb screws.

5.5 NOZZLE HEATER CONSTRUCTION

The nozzle heater supplied with the Micro-Particle source installed in the μ PP is shown in Fig. 5-9. Details of heater construction are included in the event the user needs to fabricate additional nozzle heater units. Replacement heaters can also be ordered from Phrasor Scientific, Inc. The nozzle heater unit consists of three rows of concentric heater elements aligned parallel to the nozzle tube axis. These elements are typically .020 inch diameter tantalum wire. The heater is assembled by threading the heating elements through the wire channels machined into the boron nitride heater core. To avoid breakdown of the structurally weak BN, the BN must be placed under high compression during the threading operation. The compression can be supplied by inserting the BN heater core into a small hose clamp and tightening the clamp.

5.6 RESERVOIR HEATER CONSTRUCTION

The reservoir heater supplied with the Micro-Particle source is shown in the drawing of Fig. 5-10. This heater assembly consists of a



NOZZLE HEATER

Fig. 5-9 Nozzle Heater Construction

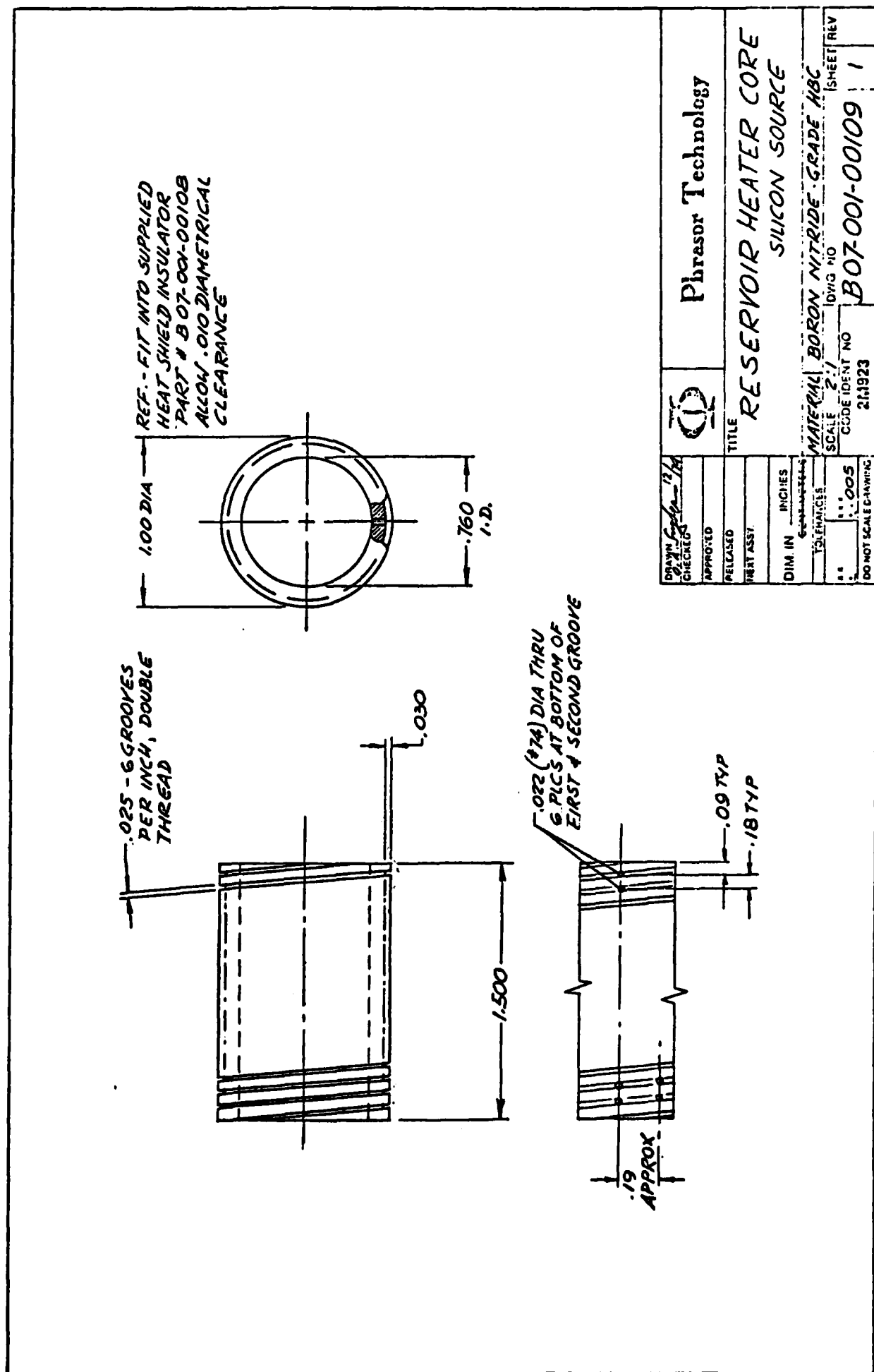


Fig. 5-10 Reservoir Heater Construction

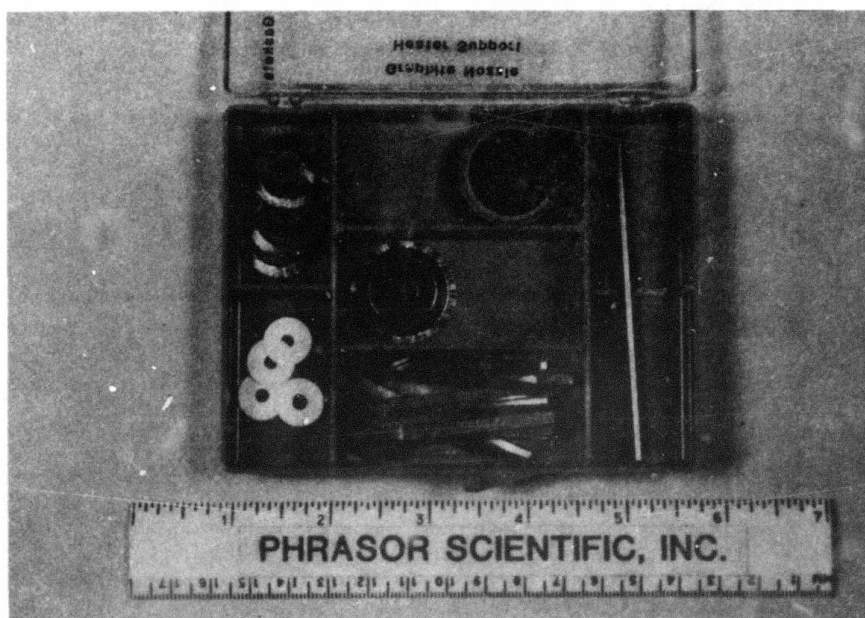
thick-walled boron nitride tube with a double-threaded groove machined on the outside diameter. Resistance wire (0.020 inch diameter) made from tantalum or rhenium is wound around the core. Rhenium is recommended over other refractory elements because rhenium has the highest electrical resistivity and thus requires less current to achieve given reservoir temperatures. Also, rhenium retains ductivity, even in the recrystallized condition, which reduces the problems of thermal and mechanical shock common to other refractory elements.

5.7 MICRO-PARTICLE SOURCE MAINTENANCE KIT

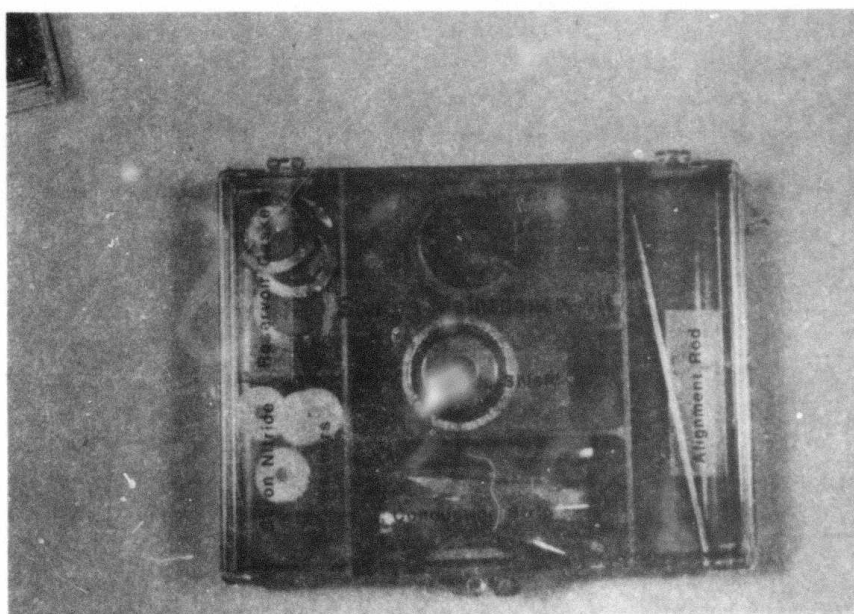
A small maintenance kit is provided with the μ PP system consisting of minor parts which are frequently used to replace spent or damaged components during routine maintenance and operation. The following is a list of parts provided in the maintenance kit (see Fig. 5-11):

1. GRAFOIL Reservoir Gaskets
2. Boron Nitride Nozzle Heater Washers
3. Graphite Nozzle Heater Support
4. Tantalum Cap Shield
5. GRAFOIL Conducting Strips
6. Source Assembly Alignment Rod

Additional kit parts can either be fabricated by the user, or ordered from Phrasor Scientific, Inc.



(a)



(b)

Fig. 5-11 Micro-Particle Source Maintenance Kit